



Bequeathed to the  
Department of Applied Chemistry  
University of Toronto

by the late

Harry Goulding Akers, B. A. Sc.,

a graduate of the

Faculty of Applied Science and Engineering, University  
of Toronto, in the Class of 1909.

Died at Yorktown, Virginia, April 21, 1917.







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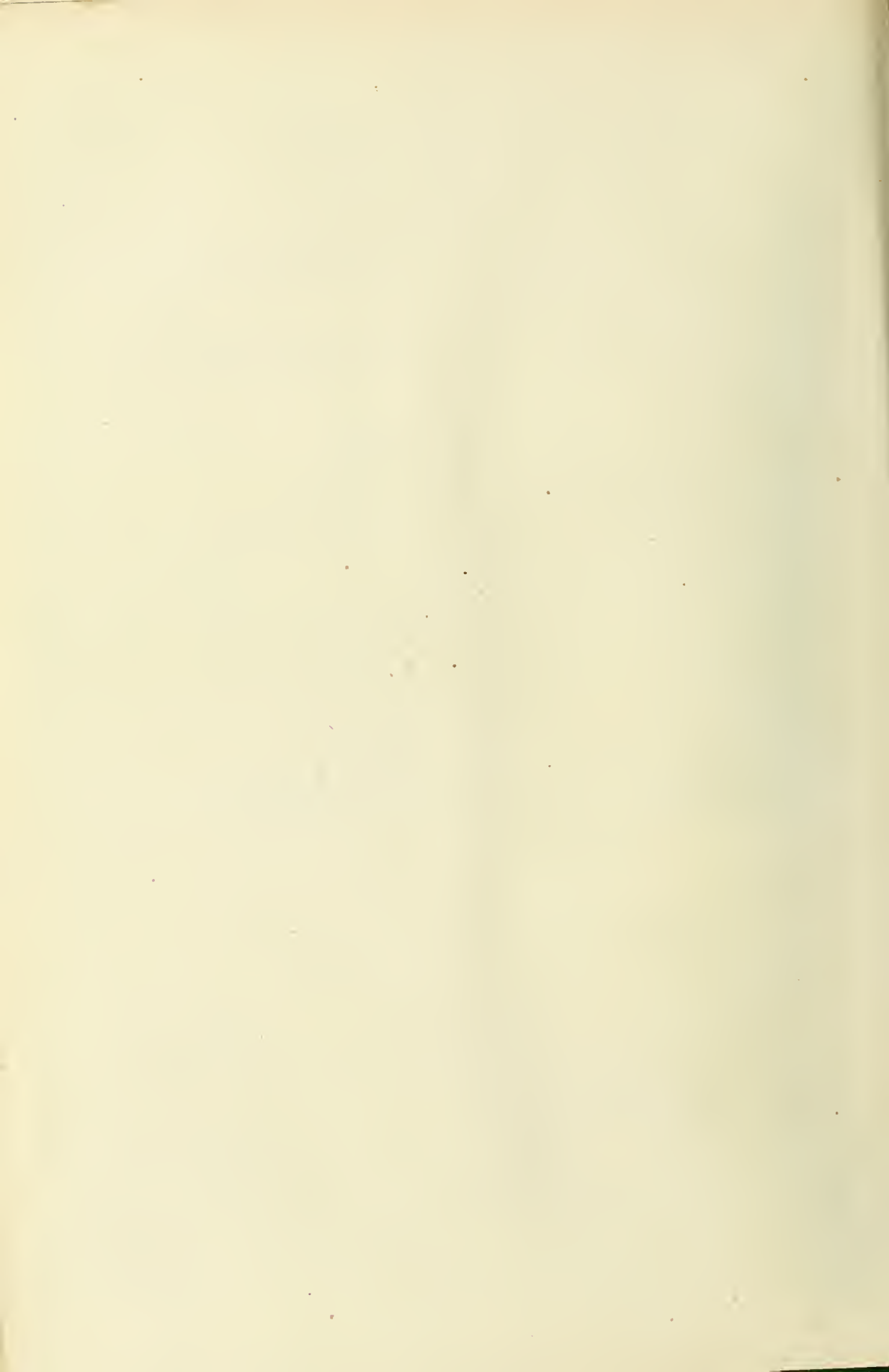


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## List of Corrections and Additions to be added to the List of Members as published January, 1884.

N.A. New Address, to take the place of that given in the January List.

N.M. New Member, or Member restored on payment of arrears.

R. Resigned since appearance of January List.

D. Deceased, of which notification has arrived since appearance of January List.

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# THE JOURNAL OF THE Society of Chemical Industry.

## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

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### NOTICES.

Members are requested to notify change of address as promptly as possible to the General Secretary and to the Honorary Secretary of the Section to which they belong in order to ensure receipt of the Society's journal.

Members will please to take notice that the Annual Subscription of £1 1s. for 1884 is now due, and should be forwarded to the Hon. Treasurer, to whom also cheques and orders should be made payable.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the first volume of the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

Numerous inquiries have been made for complete sets of the Journal for 1882. Some of the numbers for that year are out of print. Members who have not the volume for 1882, and wish to obtain it, are invited to signify that wish to the Secretary as early as possible. If a sufficient number of applications for the complete set are received, the missing numbers will be reprinted.

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The meetings of this Section are held on the first Monday in each month.

On February 4th Papers will be read by Mr. C. C. Hutchinson "On the Disposal of Sewage Sludge," and Mr. J. H. Porter "On the Porter-Clark Process."

CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE,  
 Monday, January 7th, 1881.

MR. DAVID HOWARD IN THE CHAIR.

## DISCUSSION ON THE DESIRABILITY OF UNIFORM METHODS AND BASES OF ANALYSIS, AND THE PRACTICABILITY OF ESTABLISHING AN INTERNATIONAL AGREEMENT CONCERNING THEM.

The SECRETARY notified the receipt of several apologies for absence, and of communications relating to the subjects under discussion.

The CHAIRMAN briefly sketched the history of the recent endeavours made abroad to obtain uniformity of analysis—endeavours which Dr. Lunge attributes to the suggestions made by Dr. Messel at the first meeting of the London Section of this Society. He strongly urged that no successful movement in this direction could do away with the need for individual skill. There would be an equal need for the "art," even if there were somewhat less of the "mystery." Any endeavour to dispense with high training and skill in the analyst would end in deserved failure. He put forward the importance of considering whether as chemists they could do anything to diminish the grave inconveniences caused in business by uncertainty in analysis, or whether they must leave that entirely to men of business, who, he feared, might adopt summary methods to obviate the difficulties, not always dictated by a knowledge of the scientific side of the question. There were a great number of substances, such as artificial manure, anthracene, benzol, and sugar, which were sold by analysis, which must be approximately uniform. He hoped that the attention of the meeting would be confined that evening to see how far a universal

agreement as to methods could be arrived at, and whether such agreement could be made international; and again (which was a subordinate branch of the subject), how far uniformity could be arrived at in stating results, and what could be done to promote uniformity of standards. This last was a most important point, and where again the international question was deserving of careful consideration. They might also consider whether anything could be done to discourage those barbarous systems of hydrometry which prevailed both in England and on the Continent. Another point, which was perhaps more difficult to take up, but which was a matter of as much practical importance as the chemical question, was that of sampling. This question had been carefully discussed by the chemical section of the London Chamber of Commerce, and he did not think he was over-stating the views of those who sat with him on that committee in saying that the most strenuous efforts would be used by the Chamber of Commerce to lend all the weight of its influence to secure uniformity in the like matters. Of course a practical mode of bringing it about was the promotion of uniformity in the terms of contracts. For, after all, the form of contract governed a vast number of these transactions. He thought chemists should have a voice in this important question, and should take part in the preliminary discussions which led to the settling of those forms of contract which were to govern these transactions. A large number of the members of the chemical committee of the London Chamber of Commerce were members of this Society, and therefore knew what they were talking about; but it was extremely desirable to consider at once whether chemists themselves could do anything in this matter or whether they should leave it to be dealt with entirely by commercial men, to make their own terms, as they had already done in certain special trades.

The SECRETARY then read extracts from letters from Dr. Lunge, Dr. Hurter, Mr. B. S. Proctor, and Mr. Thos. Farmer Hall.

Dr. Lunge (Zurich) wrote—

"I am exceedingly glad that the Council of the Society of Chemical Industry in Great Britain is taking up this question. It is doubtless the fittest body in that country to bring about practical results. Even if all that is desirable be not attained, good will certainly follow. The action of the chemical section of the London Chamber of Commerce is most commendable, and may materially assist the practical development of the question. I have taken a somewhat prominent part in the question of bringing about an international system of analysis and sampling of technical products, as my circular letter issued by you indicates. The November number of the Journal of the Society indicates very fully the position of affairs (page 411). The Wiesbaden meeting had gathered from my report that the time had hardly arrived for an international agreement, much as this seemed desirable, but it seemed hopeless to think of it till a general agreement has been arrived at by each country. Nobody dreams of 'crystallising' chemical analysis in a single shape, to the exclusion of all progress in that branch of science. The question, in the first instance, is one of 'technical' analysis, and secondly, of fixing uniform methods to be employed by buyers, sellers, and commercial analysts 'for the time being,' but subject to revision and replacement by improved methods from time to time. The German society thinks that this can be done within the limits of societies such as yours and its own. If an agreement be attained in England, Germany, and France, the methods employed in these countries might be compared at some international congress. This aim would be immensely facilitated, and even the national systems respectively be greatly benefited, if the chemists of these countries were in communication during the preparation of the standards for their individual countries. Such is my opinion, and I shall act upon it by placing at the disposal of your Society, or of any committee appointed purposely, all the material I am collecting for the German society's committee on analytical methods. The German society proceeded by appointing a committee on which each important industry has two members, who are to draw up a list of the products to be analysed and the methods best adapted for them. It is my task to mediate in case of difference, fill up the gaps, and draw up a report, which will be discussed at a full committee meeting. Its final report will be discussed at the next general meeting of the society, when probably an order will be given to embody the whole in a manual, which for a fixed time will be the standard for all

members of the society. The following are the industries represented on the committee; Tar and mineral oil distilling, Stassfurt products and potash salts, coal-tar products and organic colouring matters, alkali, organic acids and their salts, metals and their salts, and artificial manures. Most of the members of the committee have communicated with me, but some time must elapse before all the materials are collected. Still I can already see that very sound progress will be made in several, if not all, the branches concerned."

Dr. Lunge's letter was accompanied by a copy of the Wiesbaden circular and the questions propounded to the tar and mineral oil industries.

Dr. Hurter (Widnes) wrote—

"After the various Sections of the Society have decided that the subject ought to be dealt with, and can be dealt with, it should be brought forward by some competent person at the next annual meeting. A representative committee should be appointed, which should be placed in communication with the scientific bodies of the country, as indicated in your circular-letter of invitation. A joint committee thus formed should consider the question. No precepts nor directions should be given, and the committee should be free to take such steps as would conduce to a satisfactory solution of the problem."

Mr. B. S. Proctor (Newcastle) wrote to express his sense of the importance of the subject and his opinion that its accomplishment was but a question of time. After recommending action by representative committees, Mr. Proctor concludes—

"All that it would be desirable to attempt would probably follow in a natural course, and as speedily as convenience would allow, for it must be remembered that there will be inconvenience in the change as well as in the present want of agreement."

Dr. ARMSTRONG said that personally he had little to say, and that he came wishing rather to ascertain the general feeling of the members, so that he might be able to inform the Council of the Chemical Society, in order that they might be in the position, should occasion require, to take their part, as he was sure they would, in advocating this very important step. He could not say anything on the question of sampling, but with regard to the question of the uniform employment of analytical methods there could be no doubt whatever of the importance of carefully attending to the proviso which Dr. Lunge had laid down, that whatever method might be decided upon for the examination of any particular substance it would be necessary from time to time to revise it, in order that improvements might not be discouraged or prevented. There were many cases in which it was most important that a hard and fast line should be laid down—cases in which at present an exact analysis was impossible. In the case of tanning materials, for instance, there was no reliable process for the estimation of the tanning material, and therefore some kind of method might be adopted which was as nearly as possible an approach to the practical process. In such cases there should be a hard and fast line laid down and a prescribed process agreed upon. As time went on it was to be hoped that their acquaintance with the particular materials would increase, and that really scientific methods might be introduced. On the other hand, the chairman had said that there were certain cases in which uniformity was desirable, and he had referred to the case of benzol. Dr. Armstrong did not know whether he meant that seriously, but he was not inclined to think that it was altogether desirable that strict uniformity should be aimed at in cases of that kind, where it was possible to obtain a fairly-accurate estimate of the nature of the material dealt with. He thought if a scientific society were to advocate any particular standard at all it should be as far as possible a scientific standard. Of course if manufacturers chose to say they desired to have their material valued upon a certain basis well and good, but scientific chemists would not be placing themselves in the right position unless they proposed methods which were as far as possible scientific. There were one or two points which struck him as being of some interest in

the discussion which had already taken place on this subject at Liverpool. There was the interesting question of the difficulties which arose owing to different atomic weights being made use of. The case of manganese was quoted, and a suggestion was thrown out whether it would not be possible in many cases to devise standards which would be altogether independent of changes in atomic weights. That was a direction in which they might materially contribute to the desired end, and there were no doubt many cases in which it would be possible to take certain pure substances as standards to which the results might always be referred, and then, whatever change might take place in the atomic weights or factors of that kind, there would be no difficulty in testing. The majority of atomic weights were liable to considerable errors, and in some cases this might be of considerable commercial importance.

The SECRETARY then read extracts from letters from Mr. T. Farmer Hall, Mr. Kingzett, and Mr. Maxwell Lyte.

Mr. T. Farmer Hall wrote detailing the method of procedure by manure manufacturers in the analysis and valuation of their raw materials and manufactured products, and pointing out by tabulated illustrative analytical results, differences in the analyses of the same material, by equally eminent chemists, to the extent of  $1\frac{1}{2}$  to 2 per cent., and occasionally more, which in the case of ordinary raw phosphate represented 1s. to 1s. 4d per unit on an article costing £3 to £4. The discrepancies in the valuation of ammonia were of greater moment still. Mr. Hall continued—

"Four houses alone in London turned out more than 110,000 tons, and in the British Isles 750,000 to 1,000,000 tons per annum of dissolved phosphates and ammonia were produced in various forms, all of which were affected by uncertainty in the analytical results. . . . I do not intend to imply that in all cases the analysis was to blame for these discrepancies. The sampling in the cases of such materials as dissolved bones and bone manures is all-important, and unless correctly done no analyses would give concordant results. . . . Four firms had given him, worked out into money value, the differences which had occurred in 1882 between the analyses by chemists who in each purchase had acted respectively for buyer and seller. . . . This difference he found to be £3,076.

Seeing these differences affect seriously a trade in which there is the keenest competition, both home and foreign, and quite apart from the professional aspect of the question, an alteration is imperatively necessary."

Mr. Hall sent a copy of the suggestions which the committee of the Association of Manure Manufacturers had prepared for remedying the evils he had described, and recommending the suitability of the Society of Chemical Industry for the work, and the formation of a consultative committee.

Mr. C. T. Kingzett wrote—

"Accepting Dr. Lunge's letter as a summary statement of the matter to be discussed, then in my opinion it would be inadvisable to establish an international agreement concerning the methods of chemical analysis. Indeed, it may be anticipated that any such agreement would interpose obstacles in the way of progress, for if all analysts were to employ identical processes for the analysis of commercial products, it would create a tendency to be satisfied with the present position of chemical knowledge, and improvements would be tardily adopted by the controlling authority, even if made. Moreover, few scientific men would be content to await official sanction to improved processes of their own devising, for such formality would be felt as a degradation to intellectual efforts. It would be difficult also to provide satisfactory guarantees for the efficient discharge of duties by the controlling authority. There should be complete liberty of action left to analysts to employ such processes or methods of investigation as they may think fit, but in cases where there is likely to be any difference of opinion, it would be well for them to describe the method pursued, so that their proceedings may be open to examination and criticism. It may be safely assumed that sooner or later, as progress is made in chemical knowledge, incorrect statements made by chemists, or inaccurate methods employed by them, will be exposed. Here, as in other matters, we must chiefly trust to enlightened public (chemical) opinion. While, however, strongly protesting against the introduction of mechanical officialism into the practice of chemical analysis I admit that the various scientific societies would do useful and important service by holding conferences concerning particular methods of analysis or by conducting collective investigations. Such a course would direct collective opinion, and thus



effect, so far as it is desirable, the object which all parties who are concerned must have in view. Scientific men of trained intelligence would surely adopt those processes which have been proved to be the most accurate. Beyond this safeguard there is the further one that manufacturers and buyers may be reasonably left to take care, in their own interests, that value, and value only, is obtained or paid for the commodities of commerce. A code such as that suggested in Dr. Lunge's circular-letter would be of no value if not made rigid and compulsory, and if made rigid and compulsory, then it would unduly interfere with the liberty of individual action as prompted by individual knowledge."

Mr. F. Maxwell Lyte, replying to Dr. Lunge's questions, wrote—

"(1) I do believe that an international agreement concerning exact standards of sampling ores, raw products, and chemicals could be attained. Such an agreement would have to be based on the use of one thermometer (say the centesimal one), on the substitution of the specific gravity enunciated as such for the diverse hydrometers and areometers in use by different nations, and probably the use of the metric system of measurement. (2) I do not believe that any similar agreement could be attained in the matter of analytical methods. (3) This has been answered in 1st. (4) The best organ would be the Society of Chemical Industry. The Society of Public Analysts would be very suitable, but they are restricted to this country, whereas the English Society of Chemical Industry is very large and important, and it possesses its counterpart in Germany, and probably will possess more in other nations of Europe. (5) The agreements might be made for an indefinite time, but the affair might be placed under the control of a committee, and sub-committees to be appointed for each country, who should revise the standards where and when necessary. (6) No remarks other than already made.

Mr. B. E. R. NEWLANDS said he had been looking at the figures on the blackboard (representing differences of results in the analysis of artificial manure), which he presumed were to be taken as somewhat bad cases. He saw there two determinations of nitrogen differing by '19 and '13, results which would be considered satisfactory if they had been obtained in a scientific laboratory. With regard to the soluble phosphates, in most cases there was no greater divergence than 2 per cent. The phosphoric acid is first determined in manures, and this has to be multiplied by about two to make soluble phosphate of it, and therefore any errors in the analysis are multiplied by two in the figures shown on the board. In most of these cases there was a difference between the two analysts of rather less than 1 per cent., and therefore there was an error of probably '45 in the phosphoric acid. This was rather more than should be the case, but he knew that these cases were exceptional, and he could speak with some authority in saying that as a rule the results obtained in this industry from public chemists were very satisfactory. With regard to the best method of obtaining results which would be satisfactory to buyer as well as seller, he thought if those branches of the chemical trade which had a difficulty in the matter, particularly the manure trade, were to agree amongst themselves to form associations and appoint as arbitrator a chemist paid by themselves, and laid down that all results should be obtained by any method the outside chemist chose to employ, but should finally, if necessary, be submitted to the arbitration of the chemist employed by the association—the association being composed of buyers, sellers, and brokers—such a plan would meet all the difficulties of the case and prevent many disputes. In the sugar trade they had such an association—the "Beetroot Sugar Association"—composed of buyers, sellers, and brokers, each class being represented on the board. These gentlemen had drawn up rules which governed the form of contracts, and the price per unit to be paid above and below certain fixed standards; the sampling was done by representatives of the association; the buyers and sellers employed their own chemists; and if the results differed more than 1 per cent. the mean of the association chemist's results and the nearest outside chemist was taken. If a Manure Makers' Association were to combine with the sellers of their raw materials and the brokers, they could with the greatest ease get a sufficiently

strong body together to be able to say we will not sell or buy on any other terms than those dictated by the association, and in a short time everyone in the trade would be compelled to come into it.

The CHAIRMAN said the Secretary had not taken the figures from the worst table, which was that of raw materials, some of the discrepancies in which were as follows: Guano, total phosphates, 40'32, 36'76; bone-ash, 73'13, 78'04, etc. He did not say that some of these errors were not due to sampling.

Mr. A. H. ALLEN (Sheffield) said: Some years ago I happened to be the secretary of a British Association committee appointed to report "On the Methods Employed in the Estimation of Potash and Phosphoric Acid in Commercial Products, and on the Mode of Stating the Results." The committee submitted a series of questions to all persons likely to be able to afford information, and they received in return a number of valuable replies, including some from leading authorities. Unfortunately, however, the replies of certain leaders of the profession were conspicuous by their absence, and the replies of others were not encouraging. Thus one firm of chemists of high standing and large experience in the analysis of both potash salts and commercial phosphates wrote: "We must decline to give you the information required, as we do not think ourselves called upon to publish our methods of analysis, which we have perfected after long and careful investigation for the benefit of those who have not taken this trouble." Again, a French chemist of very high standing, replied that he belonged to that class of chemists who could not afford to work "*pour la gloire*," but who must keep their methods—their only capital—secret. The organisation proposed by Dr. Lunge would probably be far more powerful and influential than the British Association committee, but I fear the same feeling would render the scheme abortive, and that we should still have leading chemists declining to make their processes public, refusing to work by the method which might be recommended, and persistently ignoring any criticism of their results. The British Association committee made numerous estimations of potassium as chloroplatinate, and investigated with great care the merits of various processes. How far their recommendations have done real good may be learnt from the following extract from a recent letter I have received from Mr. R. Tatlock:—

"I have never heard to what extent the conclusions of the British Association committee on potash salts were acted upon by analysts in this country. In Germany, as a matter of course, they were not adopted, as the analyses made in that country, so far as they reached me, at least, were made for sellers, and I still hold that the German method gives high results with low strength potash salts, not alone from the presence of large proportions of sodium salts but from the presence of sulphate of calcium."

Mr. Tatlock's remarks touch on another phase of the question, and that is the fact that a truthful report on samples is not always desired. In a notable instance in my own experience a client said he preferred a certificate from Dr. — ignoring the presence of 2 per cent. of sulphur and lead in an iron ore, to one from me, in which the proportion of these objectionable impurities were stated. He fully admitted their presence, so that I proved my case but lost my client. I am not sure that it might not be found in practice that the establishment of national or international methods of assaying chemical products would tend seriously to retard the improvement of chemical processes on account of the diminished encouragement which independent workers would receive. We all have an instance of what I mean in the British Pharmacopœia. The objection would be greatly lessened if the revision of the analytical methods occurred more frequently than the issue of a new edition of the Pharmacopœia, but the difficulty would be to per-

suade those responsible that their previous work was not all that could be desired. I would suggest that, after inviting communications from all practical analysts on the methods of assay to be adopted, the committee should issue a report, to be published in all chemical journals, detailing the proposed processes of analysis; and then further invite and encourage criticism from all quarters, so that, in the final report, they should have before them the views of all who cared to express an opinion on the subject. Many chemists may not feel themselves in a position to recommend or prescribe a definite method as worthy of acceptance by the committee, but on the other hand may afford much practical information on the best details of working. Again, it seems to me very doubtful how far chemists should feel themselves bound to analyse by the prescribed methods. As an instance in point, I may say that I recently examined a sample of so-called "crude naphtha," which really was a mixture of benzene and other hydrocarbons, condensed from coal gas by the process described by Mr. G. E. Davis in the December number of the *Journal*. Tested by the process described by Dr. Lunge in his work on coal tar, and stated by him to have been found to give trustworthy results, the sample in question would have been reported to yield 112 per cent. of 50 per cent. benzol. Now this abnormal result was not due to the defective working or to the inaccuracy of the method recommended by Dr. Lunge, but was owing to the fact that the so-called crude naphtha was altogether different in nature from the ordinary crude naphtha obtained by the first distillation of coal tar, and with which Dr. Lunge's method of assay gives results closely agreeing with those obtained on a large scale. (I was refused information as to the nature and origin of the sample.) Here, then, is an instance in which the chemist was bound either to modify the usual method of assay or to stultify himself; and it seems to me that the prescription of a particular method of assay in contract notes must frequently be liable to similar inconveniences, however suited such a plan may be for forming an opinion on samples of the ordinary character. If carried to an extreme, the employment of official methods would, I fear, be apt to degrade the intelligent analyst to the rank of an animated machine; and, in the long run, would be sure to react on the intellectual character and general *morale* of professional chemists. Again, several well-known analysts of iron ore having disagreed as to the proportion of manganese present in Spanish manganiferous iron ore, I was requested to look into the matter. The result was that I found the ore to contain a notable quantity of zinc, which probably by some analysts had been weighed with the manganese, and by others not, according to the process they happened to employ; but in no case had the presence of zinc in these ores been previously suspected. More recently, Mr. Riley found barium in very sensible quantity in the same class of ores—a discovery which necessitated a further modification of the process of assay. Now, had an official method of assay existed at the time I refer to the presence of these unusual elements, zinc and barium, would probably never have been suspected, and if found, the prejudice and interests of the iron-ore merchant would have prevented him from paying any attention to their existence. All these facts go to prove the necessity of prescribing such methods of assay as shall be applicable to all imaginable varieties of product, and if the methods are to do this, I fear they will have a tendency to become so complicated as to prohibit their employment in the ninety-nine cases out of a hundred in which a simpler method would give practically as good results. While I should personally view the publication of official methods of assay

of products out of my own line as highly useful to me, I am not sure that I should receive with the same favour processes dealing with products with which I was familiar; and although I should probably work by the official methods under ordinary conditions, for various reasons I certainly should not undertake to limit my discretion by never departing from them. The methods of expressing results were considered in the British Association committee on phosphates and potash salts. In these cases unanimity would seem more easy to arrive at than in that of the methods of analysis, but I am afraid that any recommendations will again be opposed by the inertia of some leaders of the profession. As an instance of the difficulty of getting any recommendation adopted, I may instance the recommendations of the Nomenclature Committee of the Chemical Society. These are very imperfectly adopted even by scientific chemists, and we repeatedly find men of the first rank writing and speaking of benzol, glycerine, gelatine, etc.

Mr. E. RILEY said the result of his experience over a great number of years was this—that the getting of accurate results did not so much depend on the method of analysis as the way it was conducted. He might get any results he pleased if he manipulated differently. His assistants frequently complained of the disappointments they met with. His reply was that only by experience could those little details be acquired which were sometimes exceedingly simple, but which made all the difference between failure and success. He had perhaps as much experience in the determination of phosphorus in one direction as anyone, and it was attended with a great deal of difficulty. Taking, for example, phosphorus in iron or steel, one might get anything one liked; and so with sulphur. It simply depended on the acidity of the solution. Where the interest of the works was in question they kept the solution a little acid, and got the phosphorus low. So with the sulphur. Again, the determination of carbon had become quite a mechanical thing; and, in fact, phosphorus also. His company was now making 1,480 tons of steel a week, and every "blow" was accurately tested for phosphorus. The result inevitably was that many of the processes became almost mechanical. The analyses were done by young men who might not be very good chemists, but they had acquired accuracy by practice. He had worked these processes out, and satisfied himself that they really were accurate. His experience had been, of course, chiefly in metallurgical products. In manganese, for instance, they always employed different weights, and the analysis was done by two different assistants, one against the other, by two different methods, and was checked by Mr. Pattinson's excellent method and the ordinary galvanimetric, and the mean of the two was taken. His impression was that Mr. Pattinson's was rather low, and the other method rather high, and so taking the mean of the two was fair. He certainly thought that methods which were known to be inaccurate should be excluded altogether. He had frequently been asked to analyse certain products by certain prescribed methods, but he always refused, on the ground that he felt himself a competent analyst. With regard to the determination of phosphorus, he added a weighed quantity of phosphorus to an ascertained amount of pure metal, and set his assistants at work to determine it, and the results came out by the methods employed exactly, which was sufficient evidence that the method of analysis was accurate. He always got the credit of finding a great deal more than anybody else, but he believed he did find all that was there.

Dr. MESSEL said: All that he had heard only confirmed his previous conviction that it was perfectly practicable to arrive at definite standards. The London



sugar refiners, the manufacturers of anthraquinone, potash salts, etc., had already done so, and he could see no reason for doing the same for copper, soda, gas-liquor, phosphates, etc., in which Mr. Pattinson pointed out that much confusion now existed. If Mr. Riley laid great stress on the quantity and strength of the test solutions which we employ and on the manipulation, why cannot the conditions be stated under which the work can be carried out most advantageously? What both buyer and seller do not desire is that each chemist who samples and analyses should arrive at a different result, as is so frequently the case. That the profession of a chemist would be lowered by carrying out the suggestions before them seemed as unfounded as to assume that the profession of an engineer had been lowered by the introduction of the Whitworth gauge for screws, or the adoption of a rational standard in lieu of the old Birmingham wire gauge.

Mr. Boverton Redwood wrote—

"The provision of authoritative rules for sampling and testing was one of the primary objects with which the Petroleum Association was founded in 1866, and considerable success has attended the efforts of the association in that direction. A uniform system of sampling petroleum burning oil is adopted by the Petroleum Association, by the Petroleum Test Bureau, in Bremen and Hamburg, and by the analogous organisation, in Belgium and Holland. The chronometric and flashing-point tests are now generally adopted, and the results are in most cases strictly comparable."

After some remarks on mineral lubricating oils, and the progress attained by him towards procuring uniformity in the methods of examination, and an expression of his readiness to personally act on any committee, Mr. Redwood

"Considers it very important there should be a generally-accepted method of estimating and expressing the chief characteristics by which the value of these oils is judged."

Mr. Watson Smith wrote—

"I have just a remark or two to offer. If an international method be agreed upon, and used for testing benzols, naphthas, etc., by a fractional distillation process, then it will not be necessary that a method involving scientific exactness and accuracy should be adopted. A method giving scope for rapidity of execution, as well as a certain amount—a sufficient amount—of accuracy, may be chosen. If, however, various methods are used, then any specially-adopted method must be the most accurate, in order to avoid the odium resulting from the comparison of 'high' and 'low' methods, by choosing the method which when challenged gives a result nearest to absolute accuracy. Suppose a method adopted universally which works with expedition, but gives with a benzol testing 90 per cent. by the most exact process only 90 per cent. *e.g.*, it might be well to adopt the more expeditious method universally as the standard method—other things being equal—because as a commercial standard once fixed it would answer the purpose just as well as a minutely-exact process requiring great expenditure of time. From this point of view I think I see positive advantage in the adoption of certain international standard methods. The necessity for such standard and fixed methods in the case of benzol and naphtha testing, in which certain small working losses must occur, is greater even than in other analytical processes, because if a sample of benzol be sent to two chemists, A and B, A being a pure scientist, with perhaps ample time to spend on a more minutely-accurate and time-consuming process, and B a commercial analyst, hard pressed for time and circumscribed as to fee, A may produce a result somewhat different from B, though both men have worked conscientiously—the results being rather the products of the methods adopted."

Mr. C. T. MACADAMS said he was not prepared to speak on the scientific branch of this subject, for though he was connected with the long-suffering industry to which allusion had been made, the manure trade, he was not an expert, and therefore would not presume to offer to that meeting any observations with regard to the correct method for estimating phosphoric acid or ammonia. He left professional analysts to decide that question, but while they were discussing it he should be very glad if some uniform commercial process could be devised. No doubt there were often considerable discrepancies, and a practically-uniform method of analysis was the greatest necessity for business. A previous speaker said that soda manufacturers would not agree to a test which reduced their percentages, but the answer to that was that so long as the process was uniform the manufacturers

could adjust their prices to the process adopted. Undoubtedly chemists had cause to complain of the carelessly-drawn, and therefore imperfect, samples sent them, and he would offer a suggestion, that the sampling should, as now, be left to the buyer and seller—"mutual sampling"—but that they should draw one sample, sufficiently large, to be sent to one of the chemists named in the contract, who should divide it into three, sending part to the second analyst named in the contract, retaining the third for the umpire analyst in case of need. In that way the sampling would become more a matter for the professional analyst, who of course could not go to Blackwall, or Aberdeen, or elsewhere, to draw his own sample, but he could secure that that which he and the other chemist operated upon were practically identical.

Mr. E. W. VOELCKER said he was fully aware of the annoyance often caused by discrepancies and errors in analyses of manures, phosphatic materials, etc.; but, as Mr. Riley had stated, this was due not so much to the processes employed as to the operator, since in indifferent hands not even the very best process could be expected to give accurate results. He could not see how standard methods could be adopted when it was remembered how excessively varied in nature are the different materials which pass through the hands of an analytical chemist. To take, as a single instance, the case of phosphate analysis. Each variety of phosphate requires to be treated in a slightly different manner, according to its composition, and upon these seemingly small details depend the success and accuracy of the analysis. The question, he said, was one of experience, not of hand-books or the laying down of rules. Further, if fixed methods were laid down, the result would be to prevent the progress of knowledge amongst analytical chemists, and to tend to a perpetration of errors, even when these were known to exist. As an instance, he mentioned the case of bone ash, the phosphate of lime in which was formerly determined merely by precipitation with ammonia, the entire precipitate being reckoned as phosphate of lime, wherein it was clearly shown by Dr. Voelcker that it was in reality a mixture of the phosphates of lime and magnesia together with a varying proportion of carbonate of lime. Strong opposition to an alteration in the method of analysis, though shown to be necessary, was given by those whose interests were thereby affected, on the ground that they had been always accustomed to have bone ash analysed according to the older method. A like opposition was encountered at a later date, when it was shown that phosphates, such as Carolina, Canadian, etc., which contained fluorine and silica, required to be evaporated to dryness with acid in order to insure the expulsion of the fluorine and the rendering of the silica insoluble, thereby securing the freedom of the sulphate of magnesia precipitate from fluo-silicate of magnesia. It was complained that the new method caused a serious difference of 3 to 4 per cent. in the phosphates, but it is now generally recognised and adopted as correct. There are so many details of this kind that it is impossible to lay down rules to meet every case. The introduction of a hand-book of methods of analyses would again, he thought, tend to produce rule of thumb procedure in analyses, which would be highly objectionable.

Mr. C. NAPIER HAKE remarked that since the last meeting the Stassfurt potash manufacturers had formed a syndicate, one result of the combination being that a system more or less in force before had been confirmed, *viz.*, that all chloride of potassium should be sold according to Stassfurt analyses and weights solely. This decision might be quoted as a

prominent instance of the result brought about by differences in analyses, not by the use of different methods but simply by modification of details in carrying out one and the same method. Having had considerable experience in the analysis of potash salts, and practically tested the Tatlock method and the Stassfurt method, he was induced to say that the one employed at Stassfurt was somewhat the better—it was simpler, and less liable to error in the details of manipulation, and consequently would be more likely to insure the desired uniformity. He thought the question of a uniform system of analysis was one that could scarcely be settled between chemist and chemist, and trusted that the decision arrived at by the Stassfurt manufacturers would not prevent the bringing about of a proper understanding between the Stassfurt sellers and English buyers on lines which the Society might well suggest.

Mr. JOHN PATTINSON (Newcastle-on-Tyne) thought on the whole it was desirable that a committee should be formed to legislate upon certain matters connected with analysis of chemical products. The manure trade was one in which some fixed method was more desirable perhaps than any other of which he was aware, but at the same time there was the danger which had been already mentioned, that when once a method had been established by any authority there was the greatest difficulty in getting it altered at any future time, whatever improved methods might be discovered. If such a committee were appointed it should go very carefully to work, or it might do more harm than good to many existing chemical trades. The soda test had been frequently alluded to as a test in which unanimity was desirable. Almost ever since the establishment of the soda trade in this country it had been the practice to test soda by what was thought to be the correct equivalent of soda at that time, namely 32, which made pure carbonate of soda to contain  $59\frac{1}{4}$  per cent. of soda. That was the method Mr. Gossage arranged in 1836, and it was still followed throughout England. It made a difference of about 1 per cent. in favour of the alkali manufacturer, and it was very easy to see why that had never been altered after it was discovered that 31 was the correct equivalent, and that pure carbonate of soda only contains  $58\frac{1}{2}$  per cent. of soda. To make this alteration would at one time have meant a reduction of price of from 4s. to 6s. per ton on the product, and even at the present low price of soda it would make a difference of about 2s. per ton. In the case of soda tests, perhaps no great evil was done by retaining the old method of testing. It was well known that in England soda ash was always tested in that manner, and tables are published showing the relation of the English test to the other modes of testing. Supposing a committee were appointed, in all probability they would wish to suggest that the soda makers should immediately make the alteration to the newer and more correct test, and sell their soda by the 31 equivalent test, thus reducing the value by about 1 per cent. Was it likely they would agree to that? Unless there were a general agreement to do so amongst soda makers it would be impossible to carry out any suggestion of the kind. Again, copper ores were sold by what was known to be a very incorrect method, the Cornish assay, which was very irregular in its results, giving differences up to 2 per cent. It would be desirable, both for buyer and seller, especially for the seller, to adopt a method by which the exact amount of copper could be determined, and there are several perfectly well-known methods for doing this. But was it likely that copper smelters would follow a suggestion of this kind if made by the committee? He thought it exceedingly doubtful. Again, returning to the soda trade, his experience was that the difficulty had not

been to devise exact methods of determining the soda, but to find chemists who would do the testing correctly. The soda tests made in Liverpool were frequently from 1 to 3 per cent. higher than those made in London, on the Tyne, and in Glasgow. He had been in correspondence with most of the chemists in Liverpool, and they all told him they tested by precisely the same method as he did, namely, on the assumption that pure carbonate of soda contained  $59\frac{1}{4}$  per cent. of soda, and yet these differences existed. It seemed to him that these facts pointed to the want of a court of appeal rather than to a necessity for establishing standard methods of analysis.

Mr. ZIMMERMAN thought the men in every particular trade knew what an analysis in that trade was worth. His experience of twenty years taught him what price to put on a product sold by analysis when the buyer insisted on some certain analysis. Such was the case for many years in potash salts, until some short time ago Stassfurt potash manufacturers came to the conclusion that they would sell on one analysis. He was a merchant, not a chemist, and therefore could only speak from the commercial point of view. They all had their own remedies as buyers and sellers, and practically speaking they were not so much inconvenienced as outsiders would be, and perhaps it might be useful that outsiders should be a little inconvenienced. When he first came into the chemical trade and was a novice he made some foolish contracts by permitting certain English analyses, which gave when the goods were delivered here very different results from those by which he had purchased abroad; but he had gained experience, and he did not believe anyone in the room who had any commercial practical experience of chemicals would be inconvenienced or deceived; and he thought, therefore, it would not be to the advantage of the chemical trade to lay down any hard and fast lines for analysts.

Mr. DOMEIER said he was also a dealer in chemicals. He thought the middlemen were more interested in having correct results than anyone, because profits were small, and they had to rely on one analysis from the seller and another from the buyer. He found after a long time there was no practical difficulty at all in doing business as long as the mode of analysis were properly understood. The great mistake was that chemical analysis used to be considered a matter in which everyone had a method of his own, and which no one else ought to know. So soon as the method was understood difficulties ceased. The evil worked its own cure in commercial affairs, but he did not believe that English, French, American, or German chemists would differ materially in result if the method to be employed were fairly stated; but unless the method was thoroughly described results were liable to vary. Supposing an improved method was found out in six months' time, and was attempted to be introduced without due publication, difficulties would arise. In matters of trade it was necessary simply that the buyer and seller should agree; other matters could then be regulated by the price, and if the customer knew he must pay on an incorrect basis he must make due allowance for it.

Sir F. A. ABEL had listened with a great deal of interest to the discussion. He felt that the difficulties of the subject were very great, and there was much to be said on both sides. All would agree there would be great advantages attending an approach to uniformity in methods of work even if it were only in this country; but at the same time, what had been urged with regard to the attendant difficulties was quite correct, and any attempt to reduce chemical analysis to the purely mechanical was most strongly to be deprecated. He had had some little experience in



preparing directions for one or two almost purely mechanical methods of testing, with the special object of making them applicable with uniformity by operators who had no previous training, but only ordinary dexterity, common sense, and honesty; and the longer the experience he had of their general working the more he had become impressed with the difficulty in mastering the obstacles to their uniform application. Take, for example, the simple method of testing the flashing point of petroleum as an illustration of this. It was hoped that when one had arrived at an almost automatic test, and had laid down simple rules which could be followed with great uniformity one had done everything necessary; but when the test came to be applied by different people, and was used in different countries, it was found that it might yield in the hands of different operators results varying to such an extent that, superior as the test might have been to those previously used, it still left much to be desired. The same remarks applied to other tests of a mechanical nature which he had endeavoured to design for the examination of explosive compounds with reference to their proper purification. It was easy to give precise instructions for making a delicate test paper, to lay down the form and dimensions of the particular apparatus to be used, to prescribe the temperature and other details of the operation, but with all this it was almost impossible to get two persons to do the same thing in exactly the same way, and so he feared it would always be. All they could hope to do in arriving at something like uniformity was to arrive at some kind of general understanding as to methods of analysis which were considered the best, and then to apply themselves to carry these out honestly and to the best of their ability, disregarding as far as they could—it might be impossible to do so altogether—the bias both of buyers and sellers which would always exist. He did think that there ought to be no great difficulty in arriving at the greater approach to uniformity than existed at present in the statements of results. As to international uniformity, there were many difficulties to be got over before that could be arrived at. In the first place, we must all agree to discard our scruples, give up our draughts, and soberly drop grains for grammes. On this question he should like to hear the opinion of Sir Frederick Bramwell, who, though not a chemist, but an eminent engineer, was highly qualified to speak on this subject. Probably he would say that the difficulties in the way of the general adoption of the metric system was a formidable and a fundamental one, and he might probably be very much opposed to our relinquishing our system of weight and measurement, but they could all yield a little bit, and he believed even Sir Frederick Bramwell, though he entertained much respect and affection for our national weights and measures, would come to acknowledge the necessity of yielding to the tendency for uniformity. Theoretically he was as sanguine as Dr. Messel, but practically he realised the difficulties as fully as did Mr. Riley. It was one thing to lay down a method and another to carry it out, and the latter gentleman was quite right in saying that a man might make what he liked of a method. If we could only advance so far as to arrive at the understanding that such and such was the proper mode of working a particular method that would be a great step in advance towards uniformity.

Sir F. BRAMWELL said it was rather hard on him to be challenged on the occasion of his first visit to that Section to give a vindication of British weights and measures. He did not pretend to do it so far as chemicals were concerned, because he did not understand them, but he had a very strong opinion

as to the cumbrous nature of the simplicity of decimals. He believed if you wanted to make persons utterly incapable of mental arithmetic, so that they had always to cover sheets of paper so as to arrive at a certain result, and then find they were all wrong, because they had misplaced a decimal point, they should devote their minds from a very early age to decimal arithmetic. He had made it his business when on the Continent to notice the ability of persons there, in different stations of life, to do readily common arithmetical calculations, and he said advisedly that, compared with people in a corresponding state of life in England, brought up under the wretched system of tons, cwt., qrs., and lbs., and pounds, shillings, and pence, the ability of the Continental was not one-tenth part that of the Englishman. If you went to an English railway station, and asked for three tickets from A to B, you did not find the clerk take a piece of pencil or chalk to make the calculations. If there were three tickets at 3s. 4d. each, he had no difficulty in telling you at once they came to 10s.; but was it not the fact that you could not go to a Continental railway station and make a demand of that kind without the clerk behind the grating keeping everybody else waiting while he was making a calculation? If you went into a Parisian banking-house, and found a man at the counter, and tendered him a bill—again through a grating—you found a tedious calculation going on. At Milan he went into a bank to get a circular note cashed, and after a long calculation the clerk gave him 20 francs more than he was entitled to. He pointed out that what he was doing was wrong, but he insisted it could not be so, and he had to go over the whole matter carefully to prove it. Again, when he went through the engineering works at the Paris and Lyons Station, the officials there were very civil, and showed him the model of a locomotive with a very variable blast pipe. He asked them between what points they could vary it. A gentleman took a sheet of paper and figured away for a quarter of an hour, whilst he, seeing the drawing was to a scale of one-tenth, measured it at once; but he found when the gentleman had made his calculation the variation was something which would embrace the whole diameter of the chimney, and it turned out there was some error in the calculation—there always was an error in it. He was in Egypt for two years in connection with the sugar factory, and it was his fate to spend a great deal of time with the French officers in determining the sizes of the different parts. He used to give them mentally the result, and they said, "How do you do it?" He told them he turned the cumbrous decimal system into vulgar fractions, dealt with them as vulgar fractions, and turned the product back into decimals, and that he could do that in his head while they were figuring all over the paper. Was there a man present who could multiply  $3\frac{25}{100}$  by  $3\frac{25}{100}$  mentally? Suppose you were asked to square  $3\frac{1}{4}$ .  $3$  times  $3\frac{1}{4}$  is  $9\frac{3}{4}$ , and  $\frac{1}{4}$  of  $3\frac{1}{4}$  is  $1\frac{1}{16}$ , which makes  $10\frac{9}{16}$ . If you want to do it in decimals, by multiplying  $3\frac{25}{100}$  by  $3\frac{25}{100}$  you get  $10\frac{5625}{10000}$ ; but he could not do that mentally—he did it by multiplying  $3\frac{1}{4}$  by  $3\frac{1}{4}$ , and turning the product back again into decimals. Again, he should like to show how dissatisfied the French were with their own decimal system commercially. They did not write '001—they wrote  $1^{mm}$ . They did not take the metre and give the fraction of it, which was too cumbrous. Again, they did not use the kilogramme. If they looked in any of the shops they would see the goods marked up so much for half a kilogramme. No one said 500 grammes, or thought of saying 500 grammes, when they could say one half. Decimals were of the greatest use in their place, but he objected to being

compelled to use them whether he liked them or not. He would ask them to consider, and watch the next time they were travelling, whether there was not an absolute want of mental arithmetic on the Continent in consequence of this decimal system; whereas in England they went into a butcher's shop and asked how much a piece of meat weighing 8lb. 4oz. at 6 $\frac{1}{2}$ d. a pound was, and they would get the answer in a moment. If you wanted scientific accuracy in a great many places of figures you might use decimals, although then it was inaccurate compared to vulgar fractions; but if you wanted rapidity and clearness, take the ordinary vulgar fractions. How many things there were on the present system which were very easy. In his own business, for instance, if he wanted to know the weight of a boiler plate, if it was  $\frac{1}{2}$ in. thick it was 5lb. to the foot,  $\frac{3}{4}$ in. 10lb. to the foot, and so on—that was quite near enough. If he wanted to know the weight of round iron,  $\frac{1}{2}$ in. was 1lb. a foot, and any number of eighths could easily be obtained. You only had to square the diameter divided by .25, or multiply by 4, and you got the answer in pounds. If he wanted to know what weight of water fell on an acre he knew if  $\frac{1}{2}$ in. fell on an acre there were 101 tons. There were hundreds of similar cases which men who were at it all their lives knew which enabled him, old as he was, and having lost the faculty of mental arithmetic, to undertake to compete with any decimal gentlemen on any ordinary arithmetical problem—not on account of any ability of his own, but on account of using a plain, simple, intelligible mode of dealing with figures instead of the cumbrous system of decimals.

After some conversation as to the best mode of expressing the views of the meeting, it was unanimously resolved, on the motion of Mr. JOHN PATINSON, seconded by Mr. A. H. ALLEN—"That this meeting recommends the General Council to appoint a committee composed of chemical manufacturers, consumers, and analysts, from among the members of the Society of Chemical Industry, whose duty shall be to determine in what cases, if any, it is desirable to endeavour to obtain national or international agreement, and to report to a future meeting of the Society."

## Liverpool Section.

Chairman: E. K. Muspratt.

Vice-Chairman: Prof. J. Campbell Brown, D.Sc.

Committee:

J. F. Allen.	W. Douglas Herman.
Ernest V. Bibby.	E. Milner.
Hudson A. Binney.	E. W. Parnell.
Eustace Carey.	C. Symes.
John Hargreaves.	H. Tate, jun.
S. M. Harrison.	G. T. J. Wells.

Hon. Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be made to Edward George Ballard, Queen's Park, St. Helens.

The next Meeting of this Section will be held on Wednesday, February 6th, 1881, at seven o'clock, when the following will be the order of proceedings: (1) Discussion on Mr. Watson Smith's Paper, read December 5th, 1883; (2) Paper by Dr. Hurter, "On an Attempt to Utilise Waste Heat by means of Perkins Tubes." (3) Note on the Lancashire Soda Test, by Mr. A. Norman Tate.

UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL,  
January 7th, 1884.

MR. E. K. MUSPRATT IN THE CHAIR.

## RESULTS OF THE EXAMINATION OF THE TAR FROM SUTHERLAND'S GAS PRODUCERS.

BY WATSON SMITH.

THE quantity of tar taken was 2.5 litres, weighing as follows: First litre, 1,085grms.; second litre,

1,090grms.; half litre, 540grms.; total, 2,715grms. Specific gravity equal  $\frac{2.715}{2.5}$  or 1.085. This tar, then, unlike the blast-furnace tar and the Jameson coke oven tar oils, is heavier than water, though only slightly. Mr. Douglas Herman tells me the specific gravity he has generally found with Sutherland producer tars is 16° T., or 1.08 specific gravity. In appearance and consistency this tar is more like an ordinary or gas-retort coal tar than either of those above referred to, but it smells differently. It would appear that its being so destitute of naphthas or spirit would at least in some measure account for its high specific gravity. Were a fair proportion of these present there can be little doubt the specific gravity would be at least somewhat lower than 1.08. I think the analytical numbers will show this, but they also show that the amount of solid carbonaceous matter and matter furnishing coke and residue are very abundant in this tar. (Compare blast-furnace tar.) The tar was in a great measure separated from admixed water by shaking round in a jar, when much of the water separates mechanically, and can be decanted off. A fresh agitation then causes further mechanical separation of water and tar. In the manner indicated a large quantity of water was got rid of before the specific gravity was determined, an important matter, considering that sometimes such tars retain as much as 30 to 40 per cent. of water, and this water, containing ammoniacal salts, has a specific gravity slightly above 1. Mr. Henry Simon, C.E., of Manchester, suggested to me the advisability of using a centrifugal apparatus for separating tar and water, much in the same way as milk and cream are separated by a process already patented. However, for working on the large scale, I consider no plan is so cheap and simple as the one recommended by Lunge in his work on "The Distillation of Coal-tar," pages 97 and 101 (Fig. 4). Of course, in the case of a tar lighter than water, the running-off cock must be placed at the bottom of the still, and, in fact, the one used for running off the pitch might serve for this purpose. In any case centrifugating could not alone be a complete process of separation; it is open, besides, to other objections, and it would hence be best and cheapest to employ one simple process.

2500c.c. OF TAR TAKEN, OF SPECIFIC GRAVITY 1.085.

Temperature.	Volume of Dis- tillates.	Weights of Dis- tillates.	Specific Gravity.	Per cent. by vol. of Distillates.
	e.e.	grms.		
(1) Below 230°C.....	136	130	0.956	5.44
(2) 230° to 300°C .....	250	219	0.996	10.00
(3) 300°C. till oils solidified	362	358	0.990	14.18
(4) Oils solidifying on cool- ing, or soft paraffin scale.	231	233	265	0.996
Thick oil last coming over and at once solidifying .	—	32		
Coke .....	—	830†	—	—
Loss and Water .....	—	883‡	—	—
		2715		

\* Exclusive of water. † 30 per cent. by weight on the tar.  
‡ 32 per cent.



The results of this distillation may thus be summarised:—

Total oils obtained per cent. by weight of tar taken	=36.9
Coke obtained per cent. by weight of tar taken	=30.5
Water and loss per cent. by weight of tar taken	=32.6
	100.0

The higher boiling oils show fluorescence, but not so much as similar oils from the blast-furnace tars. Fractions 1, 2, and 3 of the preceding table of results were now mixed, and the mixture was redistilled and fractionated as follows. (Total quantity taken=748c.c. or 737grms. Specific gravity=0.985.)

	Cub. cents.	Per cent. by vol. on tar.
Below 160° C. .... (Water, 13 )	17	0.16
160° to 210° ..... (Oil, 4 )	22—A	0.90
210° to 220° ..... 51—B	2.04	
220° to 300° ..... 387—C	15.50	
300 until oils, on cooling, began to solidify	171—D	
Soft paraffin scale	69	
Coke remaining in retort	8grms.	
Loss on distillation	20grms.	

The results of the distillation may be summarised as follows:—

	Per cent. by weight of oils distilled.	Per cent. on tar by weight.
Total oils obtained	= 91.60	25.6
Water	= 1.72	
Coke	= 1.08	
Loss	= 2.60	
	100.00	

The three fractions marked A, B, and C, were at first of a light yellow tint, but they darkened on standing. The fraction distilling from 300° to the point at which on cooling solidification commenced, on cooling by a cold mixture solidified almost entirely. This portion was placed on a filter and allowed to drain, in order to separate the oil from the solid portions. The fraction marked A with the 4c.c. of oils distilling below 160°, were agitated with an equal bulk of caustic soda solution of specific gravity 1.085. The soda solution thus obtained was then a little more than neutralised with sulphuric acid, when 3c.c. of a dark-coloured oil separates, which on redistilling in a small bulk yielded 2c.c., between 170° and 190°, of a straw-coloured oil, which refused to crystallise on refrigerating and adding a crystal of phenol. The oil, however, contained phenol, as amply proved by the reactions, but contaminated considerably with cresol and other bodies. The fraction marked B, distilling between 210° and 220°, was cooled in a freezing mixture, but no naphthalene separated out. The fractions marked B, C, and D were now treated with sulphuric acid and caustic soda with the following results, those with B and C being very singular.\* Fraction B was treated with 3 and 5 per cent. of concentrated sulphuric acid and agitated, yet no acid tar was formed. After separation of the acid the oil was treated with ley of 18 Tw. without alteration of volume either of the oil or soda. Fraction C gave similar results. Fraction D, or rather the oils drained from the crystallised portion thereof, treated with 3 per cent. of sulphuric acid, gave a large proportion of thick dark acid tar, separating easily. The remaining oil (104c.c.) was then separated from the acid tar and treated with caustic soda solution, when the volume of the oils was still further reduced, leaving only 66c.c. finally. The volume extracted by

soda was then 104—66=38c.c. 100c.c. of fraction (4) from the first distillation (after the solid portion had been separated) were taken and treated with acid and soda, when 86c.c. of oil remained. The oil from this fraction (4) was very thick, and ought to be serviceable as a lubricant. The results of the treatment may be thus tabulated:—

Fractions.	Volume before treatment with Acid and Alkali.	Volume after treatment with Acid and Alkali.	Loss per cent.
B (210°—220°) .....	51	51	—
C (220°—300°) .....	387	387	—
D (300°—solidification) .....	174	66	62
IV (1st dist. Oils drained from crystallised matter) .....	100	86	14

The volume lost as acid tar from D=70c.c. Volume lost by treatment with soda=38c.c. The fractions above mentioned, after this acid and alkaline treatment, were collected and again distilled and fractionated: B, 51c.c.; C, 387c.c.; D, 66c.c.; IV., 86c.c.; total volume of oils distilled, 590c.c.

Temperature Centigrade.	Volume c. c.	Grms.	Specific Gravity.	Per cent. by vol. on tar.
Below 160°—				
Water, 22c.c. )	29.5	28.84	—	0.5
Oil, 75c.c. )				
160°—210° .....	5.5	5.67	—	
210°—230° .....	67.5	66.68	0.988	2.7
230°—250° .....	127.0	126.06	0.992	5.1
250°—300° .....	165.5	165.78	1.004	6.6
300°—350° .....	19.0	18.77	0.988	0.8
350°—solidification of oils on cooling	69.0	71.6	1.038	2.8
Soft paraffin etc.	36.0	37.25	1.035	—
Last runnings	3.5	3.6	1.03	—
Coke		28.0	—	—
	523.4	552.25		

523.4c.c. — 22c.c. water = 501c.c. oils.

Total oil obtained of that last fractionated, as above, = 84.9 per cent. On standing, a considerable quantity of paraffin scale crystallised out from the fraction distilling above 350° to the point at which solidification begins. No anthracene could be detected in the solidified distillates last received over.

PARAFFIN DETERMINATION.—10grms. of the mixture of latter thick oils were treated with 108c.c. of concentrated sulphuric acid, and the whole was heated on the water-bath. It was then cooled and poured into 500c.c. of water, when on standing the paraffin separated out as a layer on the surface of the water. It was removed carefully, dried between blotting paper, and weighed. 10grms. of thick oil gave 5.56grms. of paraffin=55.6 per cent. on the thick oil, 6.7 per cent. on the tar.

In conclusion, I beg to express my best thanks to Mr. H. Glendinning, who performed the experiments described in this paper in the laboratory of The Owens College, under my direction.

\* Since these results (as regards B and C) obtained by Mr. Glendinning appear to me at present explicable only on the ground of imperfect mixture, and as I let the opportunity pass of personally repeating the experiment, I shall take an early opportunity of repeating this portion of the process with a fresh sample.

The discussion on Mr. Smith's paper was adjourned, and Dr. ROBERT BOUCART read a paper "On the Process of Messrs. Schlieper and Baum, of Elberfeld, for Printing Indigo, including the Continuous Process for Dyeing Turkey Reds."

## DISCUSSION ON MR. W. P. THOMPSON'S PAPER ON THE NEW PATENT LAW.

Dr. HURTER spoke of the provisions of the measure in the case of two applications for patents of a similar nature, and said he did not suppose the Attorney-General or the Solicitor-General would dismiss the second application if there were the slightest difference between it and the first. Moreover, the second applicant would be given to understand how far it was the same thing, and he would surely have a right to say that there was a difference in his opinion between the two. Was there no provision for this?

Mr. THOMPSON said there was this point to be considered: Supposing the difference was very slight, the first applicant, who in nine cases out of ten could get to know what the second intended, could frame his claims so as to cover both ideas. He (Mr. Thompson) had known several cases of this kind in oppositions under the old law.

Mr. HURTER doubted if the application of one person would be seen by another.

Mr. THOMPSON said it would not, but, practically, what the invention consisted of got out in nine cases out of ten.

Dr. HURTER said, of course, every patent could be annulled after the Government had sealed it by a long and costly lawsuit; but he thought it was a very good provision of the law that a patent which was manifestly known to manufacturers to be old could be killed by a simple process. Mr. Thompson was not correct in his statement that Switzerland belonged to the countries which had a union for the protection of patents. He happened to be a Swiss himself, and he knew that the Swiss people, and particularly the chemical industries, had rejected the idea of patent laws, for, Switzerland being a very small country, could not prosper if it were hampered by patents.

The CHAIRMAN said he must first thank Mr. Thompson for his able digest of the patent law. He thought clause 9—that a complete specification should not be substantially altered from the provisional one—was a most valuable clause. He did not see that any man had a right to a patent unless he knew what he wanted to patent; and for his own part, therefore, he could not see why the clause was objected to by Mr. Thompson. If a man knew what his intention was he could state it in his provisional specification. Nor could he see any harm in the provision that the complete specification should be in accordance with the provisional one. As to clause 45, which made the Act retrospective, he thought that a great mistake.

Dr. HURTER pointed out that the Act was not made retrospective in every respect—for instance, as regards compulsory licenses and the rights of the Crown.

The CHAIRMAN said he had not had time to refer to the Act himself, and after Dr. Hurter's explanation he did not see what objection Mr. Thompson could take to clause 45.

Dr. HURTER commented upon the superiority of the new Act in regard to the point which was formerly sought to be secured by the Statute of Monopolies. What would now have to be proved in order to compel an inventor to grant a licence for the use of his patent was simply that the process was not working in the country, while under the old statute it was necessary to prove damage to trade, a very difficult and intangible matter.

Mr. THOMPSON, replying to the remarks made, said Switzerland had joined the Union in regard to

patents, designs, and trade marks, and had passed a trade-mark law, but the attempt to introduce a patent law had been defeated by the vote of the inhabitants, and could not be brought in acquisition, in accordance with the constitution of the republic, till 1892. A patent office under the Union was actually being built, he had understood, in either the city of Berne or Geneva for the use of the Union. He referred next to the fee of £100 payable at the end of the seventh year, and complained that the Board of Trade had refused to accept the sum in instalments in the case of old patents on which the £50 had already been passed before the passing of the law, but insisted upon a lump sum. In one case he had appealed against this ruling to the Board of Trade, but in vain. With regard to the retrospective nature of the Act, what he wished to dwell upon was that patents which were null and void, and were being worked by others irrespective of the patentee, could be made valid, and those who worked them could be stopped and made to pay damages. He knew people who for six years past had been infringing utterly invalid patents, and now the patentees were going to make the patents valid by a certificate of explanation, and would then attack the users of the patents and make them give up, and if the judge saw fit to decide that the original claims were framed with reasonable skill and knowledge, he could decree damages for past infringements, besides giving an injunction against the continued use of the plant erected, solely with a view of using the invalid patent, which at the date of the erection of the plant was hopelessly invalid.

The CHAIRMAN: And do you think the judge would rule that as the right interpretation of the Act?

Mr. THOMPSON said he could not help himself. He was bound to do it.

The CHAIRMAN thought such a provision ought to be altered at once, it was so palpably absurd.

Mr. THOMPSON went on to say he did not object to making the final specification agree with the provisional—he thought it should do so, and the application ought to be null and void if it did not; but what he objected to was the appointment of men to decide this question who had not scientific knowledge. The Solicitor-General or Attorney-General, up to their eyes in business as they were, had no time to attend to their duties under the Act.

Mr. EUSTACE CAREY said he was not quite clear as to Mr. Thompson's view of the retrospective operation of the Act. He understood Mr. Thompson to say that if A and B had been working an old invalid patent the owner of the patent could take steps for enforcing the validity of it. No doubt from that date A and B would have to pay royalties for the use of the patent, but he did not understand that they would be made liable for the time the patent had remained invalid, because for that period, practically, there had been no patent at all.

Mr. THOMPSON here referred to several clauses of the Act, which he read. No precise ruling upon the point raised appeared to be contained therein, but Mr. Thompson stated that in any case, whether damages could be recovered or not for the use of a patent during a period of invalidity, a patentee after making his patent right could stop its further use, and in such a case it might be that a manufacturer's plant would be stopped and rendered useless.

Mr. CAREY: He would have to pay a reasonable royalty.

Mr. THOMPSON: He can then be prevented from working the patent altogether. You will find that in clause 45.



# Manchester Section.

Chairman: Ivan Levinstein.  
Vice-Chairman: George E. Davis, F.I.C., F.C.S.

## Committee:

F. B. Bengier.	J. Jackson.
R. F. Carpenter.	S. Mellor.
C. Estcourt.	C. Schofield.
H. Grimshaw.	R. Angus Smith.
Peter Hart.	W. Vickers.
J. von Hohenhausen.	D. Watson.

## Hon. Secs.

J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

The next meeting of this Section will be held on Tuesday, February 5, 1884, at 7.30 p.m., when a paper will be read by Mr. J. von Hohenhausen on "The Separation of Benzene, Toluene, and Xylene from Crude Gas Tar, Naphtha, and Crude Benzenes." A paper will also be read by the Chairman on "A Method for the Estimation of the Isomeric Xylenes in Commercial Xylois."

MANCHESTER TECHNICAL SCHOOL, Tuesday, Jan. 8th.

MR. IVAN LEVINSTEIN IN THE CHAIR.

## GAS MAKING BY COOPER'S LIME PROCESS, WITH NOTES ON A METHOD OF GAS ANALYSIS FOR USE IN GASWORKS.

BY J. ALFRED WANKLYN.

In the course of the experiments attendant on the introduction of the Cooper lining process into gasworks, the utility of the method of gas analysis described in the *Philosophical Magazine* in the year 1881 has been illustrated in a very striking manner. Although the method has been published before, still I am encouraged by certain observations made by our chairman to offer a detailed account to the Society. As has been pointed out, there are two branches of gas analysis, which are equally logical and which are complimentary to one another. One branch of gas analysis is concerned with and measures alterations in the volume of the gas brought about by the action of reagents and by explosion. The other branch of gas-analysis is concerned with and measures the changes in reagents when they are subjected to the action of a given volume of gas. The method which I am about to describe belongs to the second branch, and consists in noting and measuring the effect on the reagent exercised by a given volume of gas.

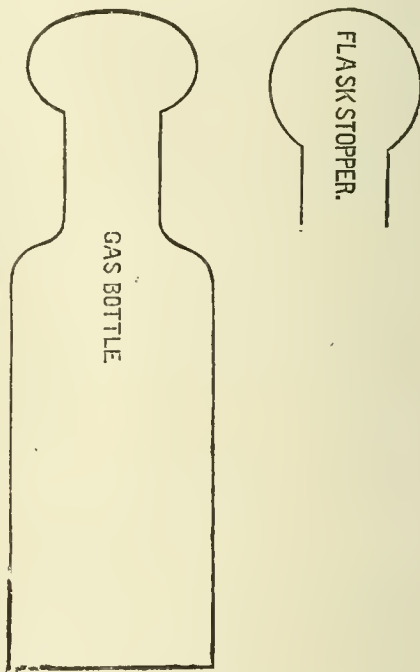
## MEASUREMENT OF SULPHURETTED HYDROGEN IN COAL GAS.

The apparatus required is—

1. A gas bottle; capacity  $\frac{1}{10}$ th cubic foot (*vide* Fig.)
2. Indiarubber tube about 2ft. in length and about  $\frac{1}{8}$ in. interior diameter.
3. A burette divided into divisions, each division holding  $\frac{1}{10}$ oz. of water. One division of the burette, or  $\frac{1}{10}$ oz.= $\frac{1}{100}$  gr.  $H_2S$ .
4. Narrow strips of lead paper, such as are in common use in gasworks.
5. A solution of acetate of lead made by dissolving 32 grains of crystals of acetate of lead in one pint of distilled water.

The manner of working will be easily understood. The gas bottle is first filled with the sample of coal gas by displacement, the mouth of the bottle being opened downwards and the coal gas being allowed to flow through the narrow indiarubber tube which is passed up inside the gas bottle. If the gas be passing at the standard rate—viz., 5 cubic feet an hour—it should be allowed to stream into the bottle for about two minutes. If the gas be passing at a much greater

rate, as is very often the case, an interval of one minute will be amply sufficient. The bottle having been filled with gas, and kept with its mouth pointing downwards, is then closed by means of the flask stopper, which has been charged with a measured volume of the lead solution. The bottle is then shaken twenty times, so as to cause absorption of the sulphuretted hydrogen. The liquid is then allowed to drain into the flask stopper. A testing is then made with lead paper, to ascertain whether the sulphuretted hydrogen has been completely absorbed. If requisite, more, lead solution is added, and the testing with lead paper repeated until the point of disappearance is reached. The number of measures of lead solution required to absorb the sulphuretted hydrogen must be multiplied by 10, and the resulting figure is the number of grains of sulphuretted hydrogen contained by 100 cubic feet of the gas. The capacity of the flask stopper is about 2oz. of water, and when very little lead solution is required water should be added, so as to provide a con-



venient bulk of liquid to insure good contact between the liquid and the gas when the shaking up is performed. Owing to the rapidity of the action between lead solution and sulphuretted hydrogen, and owing to the high sensitiveness of lead paper, the above-described operation is of a very satisfactory character. It is easy of execution, rapid, and accurate. By means of a regular system of testing, the exact degree of activity of the purifiers in gasworks may be ascertained, and in that manner most important information may be derived. The following testings performed at the gasworks in Tunbridge Wells may be cited by way of illustration. Number of grains of  $H_2S$  in 100 cubic feet of gas:—

Date, 1883.	Gas.	Gas after passing through Purifiers			
		No. 1.	No. 2.	No. 3.	No. 4.
29th November....	410	240	..	..	..
3rd December....	410	310	..	25	..
19th December....	410	350	..	140	10
21st December....	400	..	130	..	..
22nd December....	..	..	120	..	..
24th December....	400	..	..	..	..
26th December....	..	..	170	..	..
28th December....	450	280	100	..	..
29th December....	400	320	160	12	..

from which it follows that the work done by the different purifiers on certain days was: Grains of  $H_2S$  absorbed from 100 cubic feet of the transmitted gas by the purifiers—

29th November.....	170	—	..	—
3rd December.....	100	285	—	—
19th December.....	90	210	..	130
28th December.....	170	180	—	—
29th December.....	80	160	..	148

Purifier No. 1 had already been exposed to the action of gas for seven weeks on the 3rd of December, and probably at that date was as much exhausted as it can possibly become. Its persistent activity since that date is doubtless due to the revivifying action of traces of atmospheric air, which enter the gas by inevitable leakage. What may be styled the normal activity of this purifier is the power of absorbing 90 grains of  $H_2S$  from every 100 cubic feet of gas passing through it. Occasionally, as on the 28th of December, its activity suddenly springs up, owing to the entrance of a little extra air. And if we ask ourselves how much extra air must have entered to occasion the sudden rise in activity, the answer is forthcoming, viz., about 0.4 per cent. (by volume)—that is to say, one hundred volumes of the gas must have received about 0.4 volume of extra air by leakage on the 28th December. Purifiers No. 2 and No. 3 are not yet exhausted, and it will be interesting to note whether No. 2 will become absolutely inactive or whether, like No. 1, it will ultimately arrive at a condition in which it permanently absorbs 90 (or say 40) grains of sulphuretted hydrogen from every 100 cubic feet of gas passing through it.

#### MEASUREMENT OF CARBONIC ACID IN COAL GAS.

Fill the gas bottle with coal gas by displacement; add excess of baryta water in the flask stopper; shake up and allow to stand for some time; filter and wash the carbonate of baryta, which must finally be ignited and weighed. The carbonic acid is calculated from the weight of the carbonate of baryta. Multiply the weight in grains of  $CO_2$  by 1000, and the resulting figure is the amount of carbonic acid in 100 cubic feet of the coal gas.

#### MEASUREMENT OF AMMONIA IN COAL GAS.

This is best made by means of the Nessler test, if the proportion is very small. If the proportion be considerable it is best done by alkalinity. In Cooper's process of gas making, lime is employed in a manner different from that which had gone before, and with results different from that which had gone before. Up to the date of Cooper's patent lime had been used to purify coal gas after the gas had been generated in the retorts. In that manner the so-called dry lime in the modern lime purifier acts upon the gas which is passed through it; in the same manner the older milk-of-lime purifier acted; and just in the same way the oldest and most impracticable lime purifier acted, as is duly set forth in the literature of the gas industry. So far as I have been able to ascertain, the earliest mention of lime in connection with the manufacture of gas occurs in the specification of Heard, which was filed near the beginning of this century. In that specification the oldest and most impracticable modification of the lime purifiers is described. Heard used heated lime, which was carefully kept separated from the coal in the retorts, and through or over the hot lime the coal gas was allowed to pass. The method was quite impracticable, and was almost immediately superseded. Cooper's process of gas making is an entirely new departure in the gas industry. Coal pure and simple is discarded as a gas-generating material, and for it is substituted a new gas-generating material, viz., "limed coal." As may be learned from the specification, "limed coal" is

made by taking quicklime, slaking it, and thereby getting it into a state of fine division, and mixing the finely-divided slaked lime with coal. The proportions recommended are  $2\frac{1}{2}$  parts by weight of quicklime (which must be slaked) and 100 parts by weight of coal. The products arising when "limed coal" is submitted to destructive distillation in the process of gas making differs in very important particulars from products which arise when coal pure and simple is distilled.

Limed coal yields a larger fraction of its nitrogen in the form of ammonia; it also yields a slightly larger fraction of its carbon in the form of volatile compounds (tar and gas conjointly); it yields a very much smaller fraction of its sulphur in volatile forms, and the non-volatile residuum, the coke, is altered in quality, and so altered as to be improved for general purposes.

1. Experiments made in the laboratory have shown that ordinary gas coal, when limed according to Cooper's specification (instead of yielding some 6lb. of ammonia per ton of coal as in ordinary gasworks), is capable of yielding from 12lb. to 16lb. of ammonia per ton of coal carbonised. I am of opinion that, on the large scale in gasworks, an equally good result will be ultimately attained; but at present, owing to the very defective arrangement for the collection and storage of the ammonia, and also to defective mixture of the lime with the coal, a lower result has been arrived at. The utmost yield hitherto obtained on the large scale is about 9lb. of ammonia per ton of coal. But a substantial gain in ammonia, due to the liming process, has been shown by the following engineers: Mr. Trewby, at Beckton; Mr. Jones, at the Commercial Gasworks; Mr. Paterson, at Cheltenham; Mr. Wilton, at Silvertown; Mr. Eastwood, at Batley; Mr. Botley, at Wormwood Scrubs; and Mr. Dougall, at Tunbridge Wells. The highest comparative yield—i.e., the greatest difference—between the same sample of coal in its limed and its unlimed condition has been shown by Mr. Botley, at Wormwood Scrubs. Mr. Botley showed, in his experiment, that the yield of ammonia was doubled when the coal was limed. The highest absolute yield of ammonia has been shown by Mr. Dougall, at Tunbridge Wells. In the course of these trials, which have been going on during the last eighteen months, I have been strongly impressed with the inadequacy of the arrangements for the collection and storage of the gas liquor in most gasworks, and especially in old gasworks. At the old Bankside works belonging to the South Metropolitan Gas Company no gain of ammonia was registered. At the Vauxhall works, another branch of the South Metropolitan Gas Company, there was no gain of ammonia, and yet some 30,000 tons of coal had been limed. I entertain no doubt whatever that the failure to get the gain of ammonia depended on the bad arrangements which prevail at those works.

2. The slight increase in the fraction of the carbon, which assumes the volatile condition when limed coal is distilled, has been dealt with on a recent occasion, and I will pass it over without further mention.

3. The great diminution of the fraction of the sulphur, which assumes the volatile condition when limed coal is distilled, will now engage our attention. Chemists are quite familiar with the fact that sulphur present in organic compounds can be caused to attach itself firmly to calcium by simply heating the organic compound with lime; and in accordance with general chemical principles a perfect mixture of coal with excess of lime cannot possibly evolve volatile sulphur compounds when it is heated strongly. It will therefore be intelligible that limed coal should yield little or no volatile sulphur compounds when it is employed in gas making. In a preliminary experiment, in which



gas was made from limed coal, in the laboratory, so striking was the absence of sulphur compounds from the gas that the patentee recognised the gas as being fragrant rather than fetid, as is the case with common crude gas. Sulphur, as is well known, occurs in coal gas partly and mainly in the form of sulphuretted hydrogen and partly in the form of what is by gas managers termed "sulphur" or "sulphur compounds other than sulphuretted hydrogen." These sulphur compounds consist of bisulphuret of carbon and a variety of other volatile sulphur compounds which are little understood. As has been said, the main portion of the sulphur exists as sulphuretted hydrogen. I have found from 0·8 to 1·5 volume of sulphuretted hydrogen in different samples of common coal gas. The "sulphur other than sulphuretted hydrogen" in common crude coal gas, so far as I have been able to ascertain, is less than 0·3 volume per 100 volumes of gas. Both the sulphuretted hydrogen and the "sulphur compounds other than sulphuretted hydrogen" are destroyed by the application of Cooper's process, even when Cooper's process is very imperfectly carried out—that is to say, when the mixing of the lime with the coal is very imperfect the diminution of the sulphur in the crude gas has been observed. Thus, for instance, at the Vauxhall Gasworks, where the coal had been very imperfectly limed, the following observations were recorded in January, 1883:

VOLUMES OF SULPHURETTED HYDROGEN IN 100  
VOLUMES OF CRUDE GAS FROM

	Limed Coal.	Unlimed Coal
Experiment I.....	0·12	0·61
Experiment II.....	0·45	0·61
Experiment III.....	0·42	0·58
Mean.....	0·43	0·60

In addition to the evidence of a diminution of  $H_2S$  afforded by examination of the gas itself, there is other and equally conclusive evidence afforded by the prolongation of the time during which purifiers retain their activity. Thus at Tunbridge Wells the duration of the active life of an oxide of iron purifier has extended so that a life of ten days has been prolonged to much more than fifty days. In fine, the diminution of the sulphuretted hydrogen by Cooper's process is so palpable that it cannot be overlooked. Equally certain is the diminution of the "sulphur compounds other than sulphuretted hydrogen," and the diminution is palpable even in cases where the mixing of lime and coal has been very imperfect.

At the Vauxhall Gasworks there were in the early part of last year supplies of crude gas from limed coal in one retort-house and of crude gas from unlimed coal in another retort-house. Comparative testings of the gas were made, with the following results:—

1883.	Limed.	Unlimed.
20th January .....	I. 11·4	26·3
30th January .....	II. 17·1	26·3
30th January .....	III. 15·9	—
31st January .....	I. 16·4	26·3
31st January .....	II. 17·2	17·9
31st January .....	III. 16·8	—
1st February .....	I. 16·8	25·6
1st February .....	II. 11·2	18·0
2nd February .....	I. 16·1	26·3
2nd February .....	II. 16·4	27·0
3rd February .....	I. 15·6	25·1
3rd February .....	II. 15·4	25·0
5th February .....	I. 14·1	27·1
5th February .....	II. 17·3	—
6th February .....	I. 15·3	27·8
7th February .....	I. 19·7	29·5
7th February .....	II. 17·4	29·4
8th February .....	I. 16·4	28·2
8th February .....	II. 17·6	26·1
8th February .....	III. 20·5	—
9th February .....	I. 16·7	21·1
10th February .....	I. 15·6	23·5
10th February .....	II. 15·3	—
12th February .....	I. 12·8	16·7
12th February .....	II. 15·0	11·2

1883.	Limed.	Unlimed.
12th February .....	III. 11·8	15·2
12th January .....	IV. 18·0	15·2
12th January .....	V. —	21·7
13th January .....	I. 13·7	18·9
13th January .....	II. 12·8	21·2
13th January .....	III. 13·0	21·0
13th January .....	IV. 10·3	19·1
13th January .....	V. 10·6	—

On the 14th February, 1883, all three retort-houses at Vauxhall were placed under Cooper's system, and there was no gas from unlimed coal at the Vauxhall works, and the comparative testing came naturally to an end. In the above investigation the circumstances of the case precluded the employment of the referee's method, and Harcourt's method had to be resorted to. Each figure in the above statement is the mean of four or five readings. After the 15th February, when all the gas at the works was made from Cooper's limed coal, a pipe from the main in one of the retort-houses was caused to carry a portion of the crude gas to a small condenser, and then the gas went through a scrubber, and then through three boxes charged only with oxide of iron, and then it was collected in a small holder which was filled every day. The gas was tested by the referee's method, and gave the following results:—

1883.	Grains of S per 100 cubic feet of Gas.
20th February .....	11·8
21st February .....	13·5
22nd February .....	12·2
23rd February .....	13·2
24th February .....	13·6

On 26th February very little lime was added to the coal; next day no lime was mixed with the coal, and Cooper's process was stopped till 5th March, when it was recommenced and continued for many weeks. Continuing the tabular statement, the figures were as follows:—

28th February .....	21·5
1st March .....	31·0
3rd March .....	13·2
6th March .....	24·4
7th March .....	12·4
8th March .....	8·0
10th March .....	3·3

The amount of coal carbonised at that time at Vauxhall amounted to about 3,000 tons a week, and the success of the Cooper process was so manifest that even the officials of the South Metropolitan Gas Company were moved; and on March 9th, on the occasion of the emptying of one of the large lime purifiers at the Vauxhall works, the opportunity was seized to substitute oxide of iron for the lime. About a week later, a second lime purifier was refilled with oxide of iron; and on March 24th a third lime purifier was charged with oxide of iron, and from that time forward, for a considerable period, no lime other than that employed in the Cooper process was used at Vauxhall. As I have already mentioned, the liming of coal as carried out at Vauxhall was very irregular and imperfect. Since that time the Cooper process has been put in operation under less unfavourable conditions.

By the aid of the admirable machine which the gas industry owes to the eminent and accomplished engineer whose services the Corporation of Manchester are about to lose, a good and regular admixture of the lime with the coal has become practicable. And at the Tunbridge Wells Gasworks—notwithstanding that the coal is rather highly sulphurous, containing 17 per cent. of sulphur—an average of 3 grains of sulphur per 100 cubic feet of gas has been maintained for three weeks. Not only has the lime purifier been abolished at Tunbridge Wells, but so altered is the function of the oxide of iron that the manufacture of gas in Tunbridge Wells has been correctly designated by Mr. Spice as an operation in

closed vessels. With the sulphur reduced to 3 grains per 100 cubic feet of coal gas, and with purifiers which require changing only once or twice a year, the gas industry enters upon a new era.

## DISCUSSION.

Mr. WEST (gas engineer to the Manchester Corporation): It is a question of much importance to gas engineers as to which is the best process of purification, the one adopted by the London companies or the Cooper process. The latter process has not been adopted on a large scale throughout England, inasmuch as what can be made a success in one place cannot be achieved in another, owing to the difference in the quality of material used. The quality of the cannel and coal used in the south is of such a nature that the lime will mix with it more thoroughly than it will in Manchester. The question might arise whether the addition of the lime in the retorts is better than placing it in the purifiers for the removal of the sulphur compounds. In London they are under stricter regulations, and they have a larger number of purifiers, and they use oxide of iron with lime; and although we are not subject to these restrictions in Manchester our purifying plant is very extensive, and by the adoption of the lime process we could remove the sulphur compounds. Of course we have many difficulties to contend against in a large works that they have not at Tunbridge Wells, and had it not been for these difficulties we might have been trying the Cooper process at the present time, but seeing that we have so many different kinds of cannel and coal which give different results—some a larger quantity of ammonia than others—no test would be satisfactory unless carried on on a large scale, and as gas engineers we only require some definite proof of the advantages claimed to induce us to adopt this particular process. For my own part I do not doubt but there will be some drawbacks. While there is an increase of ammonia and a reduction of the sulphur compounds, the question arises, How does it affect the coke? We are informed that in the south there is no detrimental effect, but there they have about 5 per cent. of ash, while in some of our materials we have 20 or 30 per cent., and this again increases our difficulties. By the application of lime in the retorts we should lose the sulphur, which is now taken up in the oxide purifier, and this would be a serious loss. We received £4,000 last year for spent oxide, and this would be lost if the sulphur were taken out. It was a question which was the most economical of the two systems referred to. They had been told that the Manchester gas contained from 20 to 40 grains of sulphur to 100 cubic feet of gas, but he did not attach much importance to that. It was published in the gas committee's reports annually, and it varied considerably every day on account of the different qualities of material used. In reference to the complaints made as to the danger to health and the injury done to books and other articles, we have the evidence of Drs. Russell and Ogden, of the South Metropolitan Gasworks, that these impurities are not so injurious as they are made out to be, and it has been proved that the damage done to the books in the London libraries was caused by heat from a flue, and the destruction which is attributed to the impurities of the gas may be traced to similar causes. The purification of gas is a question of pounds, shillings, and pence, and we as gas engineers are willing to remove the impurities if the public will pay the cost.

Mr. HUNTER (gas engineer to the Salford Corporation): The difference in the percentage of sulphur in the Manchester and Salford gas, as compared with London, was to be accounted for in a measure from

the fact that the London companies had the best known process of purification, but the plant in other respects was very similar. I do not know exactly whether Professor Wanklyn gave us figures for comparison, but I think we should take the best known methods of abstracting these impurities. Will Prof. Wanklyn kindly tell us now what method of comparison he employed?

Professor WANKLYN: During the time the gas was limed at Vauxhall the result was very much the same as that obtained by the most perfect system of lime purification, and when this system is resorted to they can reduce the sulphur to something like 12 grains, but we profess to keep it down to 2 or 3 grains, and we have done this in retort-houses carbonising 3,000 tons of coal.

Mr. HUNTER: I think that puts it very clearly. First, we have the old method of purification by oxide and lime or the two combined, the second is the one adopted by the London companies, and the third is that advocated by Professor Wanklyn. In the two last processes the sulphur impurities are very much the same, but in justice to the Cooper process we are bound to admit there is an increase in the amount of ammonia and the volume of gas produced. With regard to the experiments in Salford, whether we did our duty or not, or dealt with the matter in an imperfect manner, I am not prepared to say, but we did not obtain satisfactory results. We had an increased amount of ammonia, and the purifying boxes ran longer, but we did not obtain an increased volume of gas. While admitting these two advantages, the addition of moisture to the dry coal seemed to be detrimental to the yield of gas, notwithstanding the professor told me that 2½ per cent. of lime moistened did not make any difference in the yield of gas; and taking this into consideration there is not very much to be gained by the process—in fact, it is more a loss than a gain. Our manner of dealing with it may be different to that carried on at Tunbridge Wells, but every facility was offered to the professor in making the experiments. I think there is much needless alarm as to the impurities in gas, and I believe it is impossible to prove they are injurious to health or materials.

Mr. R. F. CARPENTER said he would like to know whether there was any loss of illuminating power by the application of the Cooper process? Could Professor Wanklyn also say how the sulphur was combined with the lime in the coke, as sulphide of calcium was liable to give off sulphuretted hydrogen, and how would the moisture used for slaking the coke affect this?

Mr. H. GRIMSHAW: My first acquaintance of the large amount of sulphur in the gas was in finding drops of vitriol on my globes and the damage done to my books by the sulphuric acid. I think these impurities might be got rid of by utilising the gas profits, which at present were appropriated by the Corporation for street improvements, in the adoption of one of the processes for purification which had been referred to. I should like to know what was the effect of the lime on the coke as a commercial article, and what are the respective quantities of lime used in the Cooper process and the ordinary lime-purification process.

Mr. MELLOR: It has been stated that there was something like 40 grains of sulphur in the Manchester gas, and Professor Roscoe said some time ago that experiments made at The Owens College gave 27 grains. It was very desirable to know what were the facts. With different kinds of coal the result must vary.

Mr. SCUDDER: I made the experiments at Salford and found that the addition of slaked lime lowered



the temperature so much that the yield of gas was reduced from 9,000 to 8,700 cubic feet. When it was drawn from the retort the water from the coke was found to contain sulphide of calcium. I cannot concur in Professor Wanklyn's statement that in the use of lime the coke was not deteriorated. Two or three per cent. of lime, in addition to the amount of sulphur arrested—say, about 5 per cent.—added to the already large amount of ash, is a serious matter. I have made many experiments on the amount of sulphur in Salford gas, and the lowest result has been 24 grains of sulphur to 100 cubic feet of gas.

Mr. WATSON SMITH: We have tested the gas at the Owens College during the last few days, and the result has been 31 grains of sulphur in 100 cubic feet of gas.

Mr. GEORGE E. DAVIS: I simply rise, Mr. Chairman, to enter my protest against the statements which have fallen from the lips of the gas engineers this evening, that there has been much needless alarm as to the sulphur impurities in gas. I wish to say that, as yet, we, as householders, are not sufficiently alive to the very deleterious effects of this sulphur impurity. The Government has long ago recognised the dangers to which it may be subjected from this source, and has limited the amount which the London companies may not exceed, except under heavy penalties. The Government also, recognising the danger of allowing an unlimited escape of sulphurous acid into the outside air, has legislated in this direction also, so that manufacturers are not allowed to emit above a certain quantity of acid gases, sulphurous acid being one of them.

#### REPLY.

Prof. WANKLYN: If I may be allowed, at this late hour, I shall omit to reply to several of the questions which have been asked. With regard to the cost I have gone very carefully into the figures, and an eminent gas engineer has examined them, and he assures me that the statement is very favourable to the Cooper process. As to the question of the condition of sulphur in coke, no doubt some of it exists as sulphide of calcium, but there is some free lime also, and it is very probable that lime and sulphur may exist side by side. The quantity of lime used in the Cooper process is barely equal to the sulphur. It has been stated that the coke is damaged by reason of the lime mixed with it, and that the coke so used in furnaces is liable to destroy them, but I have had experience on over 30,000 tons of coal with the liming process, and I have found but little damage to furnaces in using this coke. It is certain that difficulties may arise in the practical application of the Cooper process, as every works is carried on in a slightly different manner. There is no doubt much difficulty in getting workmen to believe in any new process, and it has been my experience in every case that within 24 hours after the introduction of the Cooper process some difficulty has been found to exist. In works where the workmen are well looked after, and the manager interests himself in the process, these difficulties are soon overcome, but in other cases, where the workmen are left to their own resources, the process is condemned at once. In Salford I am not surprised that the results were not quite so favourable as at Tunbridge Wells, as the coal was used in large lumps, and a perfect admixture of the lime with it was practically an impossibility; but where the lime can be thoroughly mixed with the coal there is no doubt that the result of the process will be satisfactory. Where the Cooper process is used, it may be said that the purification of the gas is practically accomplished, in closed vessels, for the charges in the purifiers last much longer than when used in the ordinary way, and

we have found that the use of lime purifiers can be dispensed with altogether. The small percentage of carbonic acid does not seem to have much effect in reducing the illuminating power of the gas. It seems to be the opinion of the eminent gas engineers we have present this evening that the presence of the sulphur compounds in the gas is in no wise detrimental to health. When I first studied the subject I was inclined to think that the small amount of sulphur was a very slight matter, but I think differently now, especially as I have found that the quantity may be easily reduced to below 10 grains per 100 cubic feet, and I am sure everyone will agree with me that it is the duty of gas manufacturers to supply as pure an article as possible. The atmosphere of the underground railway in London is simply unbearable on account of the sulphurous acid which is mixed with it.

### Newcastle Section.

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W. C. Reid.

J. F. Stark.

T. W. Stuart.

Hon. Sec.: J. T. Dunn, M.Sc., 29, Oxford Street, Newcastle.

Meetings—Session 1884.

February 7th, 1884.

March 6th, 1884.

### GENERAL MEETING AT THE COLLEGE OF SCIENCE, NEWCASTLE, January 10th, 1884.

MR. B. S. PROCTOR IN THE CHAIR.

The CHAIRMAN: As usual at this meeting, we have gathered together a collection of apparatus, etc., for the inspection of the members, but we have in the first place a very important matter to discuss. The letter of Dr. Lange, published in the report of the meeting of the Liverpool Section, in the Journal for November, has raised the question of international methods of sampling and testing commercial products, and the Sections have been invited by the Council of the Society to discuss the desirability and practicability of establishing such international agreement on methods of sampling and testing. I believe that we are all agreed that the discrepancies which frequently occur amongst the reports of different analysts testing the same substance is a wrong thing, and we are agreed also as to the importance of taking steps to remove such a state of things. I believe, however, that this can only be accomplished in the lapse of a long course of time, and I doubt whether at present we are able to do much in the matter beyond endeavouring to obtain a full expression of opinion from all those concerned. The discussion at Liverpool has already brought to light many instances of widely-differing results got from different methods of testing the same substance, and several expressions of opinion that the contract notes between buyer and seller should state by what mode of testing the value was to be determined. If a committee was appointed no doubt its first duty would be to gather from different trades and different districts information as to how far these expressions of opinion at the Liverpool meeting were supported. The decision of such a committee would soon acquire very considerable influence, and in all probability would ultimately have the authority of an umpire in all disputed cases. I have

no doubt that to-night we shall have further illustrations of the difficulties and inconveniences which our present want of system causes in many industries. Mr. Pattinson has promised to open the discussion with a short paper, which I will now call upon him to read.

## INTERNATIONAL METHODS OF SAMPLING AND TESTING.

BY JOHN PATTINSON.

THE General Council of the Society of Chemical Industry, at their meeting on the 23rd of November last, passed the following resolution: "That this Council approves of and adopts the resolution of the Liverpool Section upon methods of international sampling and testing, and recommends it to the consideration of the various local Sections of this Society, with the view of eliciting an expression of their opinion upon this important subject." It is in accordance with this recommendation that the matter is brought before your notice this evening, and I have undertaken to open the discussion by a short paper.

As those of you who have read the Society's Journal for November last are aware, this question of attempting to bring about an international agreement as to methods of sampling and analysing ores, raw products, and chemicals was first brought forward by Professor Lunge, of Zurich, at the suggestion of the German Society for the Promotion of Chemical Industry. Dr. Lunge addressed a circular letter, the purport of which has already appeared in the Journal, to the representatives of the most important chemical industries, and to commercial analysts, in England, Germany, and France. It does not indicate a widespread desire for change in the present state of things, nor augur well for the chances of success of any scheme for effecting an international agreement, that only 10 replies were received to the 40 letters sent to England, 3 to the 43 sent to Germany, and 7 to the 43 sent to France, or 20 replies to the 126 inquiries. It is obvious that any attempt to produce uniformity in these matters, in order to be successful, should secure the general adhesion of the parties most concerned in the various countries.

I think most of us will admit that in the abstract, for the purposes of facilitating calculations and of avoiding misunderstandings, it is desirable to have an international agreement as to uniform methods of analysis for determining the commercial value of products, and as to uniform methods of expressing the results of analysis. It is, for instance, desirable that methods of analysis should be used by which the same sample of manure wherever tested should indicate the same amount of soluble phosphoric acid, and that a unit of soda, chlorine, iron, manganese, lead, or copper should mean the same thing in all countries.

It is on the questions of how far it is desirable and how far it is practicable to bring about an international agreement on these matters that there will be differences of opinion. When it is considered that in some trades there are usages in the matter of analysis, to alter which would probably seriously reduce the value of the commodities, and when it is remembered that analysts have their own prejudices and predilections in favour of certain methods of analysis they are in the habit of using, and which they would be unwilling to give up in favour of methods which they believe would give less correct results, it will be seen that there are many difficulties in the way of arriving at uniform international methods of analysis.

In the purchase of copper ore, for instance, it is a

"trade" custom to determine the amount of copper these ores contain by what is known as the Cornish assay. It is well known that this assay does not indicate the whole of the copper the ore contains, and that it is moreover very irregular and uncertain in its results. Will the committee which may be appointed on the question decide that in future all copper ores should be analysed by methods which are known to give exact and uniform results? And, if so, will all the copper smelters agree to purchase by the method prescribed by the committee? Again, in the chief chemical industry of the kingdom, and one in which this district is largely interested—the soda trade—there are some trade usages which prevail in the mode of testing and of stating results, all of which affect the commercial value of the commodity. For instance, from the earliest period in which the Le Blanc process of making soda has been carried on in England it has been the custom of soda manufacturers to sell soda for commercial purposes by what is known as the English test. This test is based on what was at one time thought to be the true combining weight of soda, viz., 32, by which pure carbonate of soda is assumed to contain 59½ per cent. of soda. It is now considered that 31 is the true combining weight, and that pure carbonate of soda contains 58½ per cent. of soda. This is a custom perfectly well known by both buyer and seller, and alkalimetric tables showing the relation of the English test to other modes of testing are in almost every handbook of chemistry and chemical analysis in the English language. It cannot, therefore, be said that any purchaser is misled or deceived by the test. It is easy to see why the change from the old-established mode of testing to the more theoretically correct new one has never been made in this large trade. To do so, at one time, when soda was more valuable than it is at present, meant reducing the price of soda ash by four or six shillings per ton, and at present would mean a reduction of two or three shillings per ton in the value. Will the proposed committee decide that the English soda makers must henceforward sell by the more theoretically correct test? If so, can the soda makers recomp themselves for the change by increasing the price of soda ash? I know some soda makers think this is impossible. If this is the case, are the soda makers likely to agree to reduce the value of their product in this manner, at a time when their trade is depressed and struggling for existence? Under these circumstances, is it likely that all commercial analysts will fall in with the recommendations of the committee and test by the new international method?

There is another trade usage in connection with the testing of soda ash which should be dealt with by any committee which may be formed. I mean that relating to fractions of percentages. It is the custom to buy and sell soda ash by whole units, and to discard fractions. At one time it was the custom to discard all fractions of units, so that a soda ash of 51.9 per cent. of soda was bought and sold as only 51 per cent. More recently it has been the practice of commercial analysts in Liverpool to give a certificate that a sample contains, say, 52 per cent. of soda, when the test only indicates 51.5 per cent., or any fraction above this up to 52.4 per cent. Other analysts in London certify that a sample contains, say, 51.1, 51.2, and so on as found up to 51.5 per cent. of soda; but if it contains 51.6 per cent. a certificate is given that it contains 52 per cent. It has been my practice during the last twenty-five years to show all the fractions on the certificate, and to test by the English test, stating this on the certificate, believing that the old custom of testing by the English test and that of giving away all the fractions should stand or fall to-



gether. It is desirable that this point should be dealt with in the case of any proposed change.

Before leaving the subject of soda tests I should like to refer, in passing, to Dr. Hurter's remarks made at the Liverpool meeting on the differences between what he calls the Lancashire and the Newcastle soda tests. Dr. Hurter accounts for certain differences found to exist between tests made in Liverpool and those made on the Tyne by assuming that the higher Liverpool tests were made with test acid, based upon Fisher's and Dalton's tables of equivalents, published respectively in 1802 and 1808. This is an ingenious theory, but it is not borne out by facts. Many years ago I was in correspondence with most of the analysts in Liverpool, from whose laboratories the high tests emanated, and they assured me that they tested with test acid made in the same way as my own, viz., on the assumption that pure carbonate of soda contains 59½ per cent. of soda. There is, moreover, printed evidence that so far back as 1836—ten years after the first soda works was started in Lancashire—the same test was used. The late Mr. Wm. Gossage, who has been styled the father of the English soda trade, published in the *Chemical News* of the 17th of January, 1863, a paper entitled "A Method of Assaying Rough Soda (black ash) for the use of Soda Manufacturers, arranged by W. Gossage in 1836," and which Mr. Gossage says he still continued to use in 1863. In this method the test acid is directed to be made on the assumption that 32 is the equivalent number of soda, or that pure carbonate of soda contains 59½ per cent. of soda. There is therefore no doubt that what I have described as the English test is the test which has been in use in Lancashire as well as in other parts of England almost ever since the soda manufacture has been in existence. The differences in the Liverpool and Tyne tests exist, although there is, and has been, a theoretical agreement as to the method of testing, and the same difference would probably occur even although a committee might decide to recommend another method of testing. This points to the need of a court of appeal rather than to the formation of a committee to recommend methods of analysis.

Perhaps the case in which there is most need for a defined and prescribed method of analysis is that of the estimation of phosphates in manures. This has been and still is a fruitful source of dispute. So far as my experience goes, however, the cases of dispute are not now so numerous nor the differences so great as they formerly were. This is probably partly owing to the buyers and sellers learning to know where they can get high tests and where low; or it may be, and I hope it is, owing to better methods of analysis being adopted. I do not know of many other cases in which the differences of analysis are so great and the disputes arising therefrom so numerous as to call for any special legislation from a central body.

From a commercial standpoint we see arguments of weight both for and against the adoption of international methods of analysis. However much we may think it desirable to be agreed upon methods of analysis which shall be constituted the standard methods for the respective articles of manufacture or trade to which they refer, so that in the hands of two equally-skilled chemists the same results may be obtained, we see also that there are objections to the establishment of such standard methods, which in many cases render it a very delicate question to deal with, involving the whole monetary basis on which a trade has been founded and now exists.

Apart from this merely commercial point of view, I can also see other objections to the scheme which seem to me worthy of consideration. Speaking as a scientific man, I fear that one result of prescribing

standard methods of analysis would be to limit original research. How often in the history of chemistry have methods of analysis, believed at one time to be correct, been proved by the knowledge gained from research to be inaccurate, and have had to be given up, others being adopted, which again in their turn have been abandoned? The desire of chemists should always be to seek continually for better methods of analysis. If standard methods be prescribed, it must to a very great extent remove the incentive to original research, and a class of chemists would arise who would become mechanically skilled in these methods, but who would do nothing to further our knowledge of science. I agree with Dr. Lunge in thinking that by the co-operation of manufacturers and chemists new and admittedly better methods might be adopted as standards from time to time in the place of former standard methods, provided such new methods are discovered; but what I fear under the proposed scheme is that when fixed standards have once been established the inconveniences and obstacles in the way of any further change would be so great that chemists would be contented to go on using the prescribed methods, and cease to strive after more perfect ones.

As to standard methods of sampling, I am inclined to agree with Dr. Hurter that these can only be prescribed very generally, and that in most cases it will be best to leave the sampling to the judgment of the buyers and sellers or their agents.

I attended the meeting of the London Section of this Society on Monday last, when this question was discussed, and at which the representatives of various trades were present. The general feeling seemed to be, that whilst theoretically it was desirable to have an international agreement on methods of analysis, the difficulties in the way were so great that its attainment was impracticable. Amongst other cases that of potash salts was mentioned. Mr. Allen, the reporter for the committee appointed by the British Association, some years ago, to endeavour to arrive at a uniform method of testing potash salts, stated that although a method which was supposed to give the most exact results had been recommended by the committee, yet no change had been made in the methods of analysis by which these salts were bought and sold. It was also stated that the Association of German Potash Manufacturers had recently passed a resolution to sell only by the higher tests of the German analysts. Mr. Newlands mentioned at the meeting a method adopted in the sugar trade for settling any differences arising from analysis. Sugar is bought and sold on the arrangement that if the analyses of buyer and seller differ to a certain amount the sample is sent to an agreed umpire, whose decision is final. I venture to think if this plan were adopted by the manure trade, or any other trade inconvenienced by the differences found in analyses, the disputes would be satisfactorily settled, and the differences arising from different methods of analysis would very soon disappear.

From what I have said above it will be seen that unless the functions of any committee which may be appointed to deal with this subject are carried out with great care and judgment more harm than good may result from any attempts to change the present state of things. I am disposed to think, however, that in a Society like ours, including in its members many manufacturers and consumers of chemicals, as well as analysts and commercial men, a committee may be formed, into whose hands this matter may be safely placed. This committee should endeavour to decide in what cases, if any, and under what circumstances, it is desirable to take action, with a view to

obtain international uniformity, and report to a general meeting of the Society, and I am prepared to move a resolution to this effect.

Mr. Pattinson then moved the following resolution, which was seconded by Mr. W. W. Proctor: "That the Newcastle Section recommends the General Council to appoint from the members of this Society a committee composed of chemical manufacturers, consumers, and analysts, whose duty it shall be to determine in what cases, if any, it is desirable to endeavour to obtain national or international agreement on chemical questions affecting the value of chemical products, and to report to a future meeting of the Society."

#### DISCUSSION.

The CHAIRMAN: Mr. Pattinson has set before us very efficiently the difficulties which lie in the way of international agreement; still we should not regard these difficulties, without further discussion, as insuperable. I am inclined to think that in many cases, if buyer and seller were to adopt the practice which Mr. Pattinson has instanced as holding in the sugar manufacture, of stating in the contract the method of analysis to be used, and in the event of disagreement between the analysts referring the matter to the final decision of an umpire, these differences would in the natural course of things gradually disappear.

Mr. JOHN MORRISON: I believe the manure trade is the one which suffers most at present from analytical discrepancies. But there are great difficulties in the way of introducing new methods, or uniformity of method, for the relations amongst chemists, manufacturers, and buyers and sellers would for a time at any rate be upset. At present, for instance, London tests are, as a rule, higher than provincial results, and it would be difficult to get manufacturers to agree to any lower tests, for selling at any rate. In the soda trade the matter is not so serious. Errors exist, and the difference between the Liverpool and the Tyne tests, for instance, is a thing which none seems ever able clearly to understand or explain; but here the discrepancies are tolerably uniform, and can be allowed for. I believe, however, that a standard method of sampling should be enforced, for I know that sampling is frequently done at present in an extremely loose manner. In the soda trade you, as a rule, buy and sell by the actual tests of the analyst's sample; but in the manure trade you guarantee a certain percentage, and practically, in order to allow for possible differences of analysis and of irregular sampling, you have to make your goods up to something considerably over the guaranteed strength. This is another source of irregular loss to the manufacturer.

Mr. MOULT (Langdale's Chemical Manure Company Limited): This subject has been often discussed by the Manure Makers' Association, but nothing definite has come of it. We have had interviews with Dr. Voelcker and other London analysts; and by some of the manufacturers it has been suggested that we should fix a method of analysis, and buy and sell only by that method. At present we do not know what method is used in an analysis, but we get to know gradually, if we send samples to a certain chemist, how his results will come out. The differences are not nearly so great as formerly, but still there are differences of as much as  $\frac{1}{2}$  per cent. of phosphoric acid in an ordinary 25 to 30 per cent. soluble superphosphate. I endorse Mr. Pattinson's resolution, but I certainly think it would be desirable to have fixed methods of analysis, as far as possible, for commercial purposes.

Mr. JOHN GLOVER: I do not know that I can add much to what has been said. I am in favour of

Mr. Pattinson's resolution. I do not think that there would be so much danger of stereotyped modes of thinking, etc., of stoppage of original research, from the adoption of fixed methods of analysis, as he seems to fear. I do not see why that should happen in analytical chemistry more than in any other science. In my opinion the chief difficulty in the way is in educating merchants and manufacturers to see the advisability of fixed methods of analysis. If Associations like the Iron and Steel Institute, the Manure Makers' Association, and Alkali Association could be brought to see the necessity for a fixed method, then the Scientific Societies, the Chemical Society, the Institute, and this Society would be able to advise them as to the choice of the best method in each case. If only there is a call from the great majority of buyers and sellers for the best method of testing in any particular case the objections of individuals to the method will have to stand aside. I have often found differences in analyses (especially in alkali and bleach testing) explained by attention and value being given to the moisture in the sample at the time of weighing. As regards sampling, I would point out that much loss is often caused to buyers of ores, such as pyrites, manganese, etc., through the unavoidable drying of the sample by the heat engendered in the breaking up and pounding necessary to obtain it.

Mr. STARK: I quite agree with Mr. Pattinson's proposal of a committee to consider the matter. The first step must be to get a clear statement of what it may be considered desirable to do, and to fully discuss the difficulties in the way. On the question of sampling there must always be a good deal of liberty and discussion allowed. Any attempt to draw a hard and fast line would lead, I am convinced, only to further difficulties.

Mr. DAVIDSON: I came here to-night, not with the intention of offering any remarks, but of obtaining, if possible, some satisfactory explanation of a matter which from time to time has perplexed me, viz., the great difference which exists between the Lancashire and Tyne tests of soda ash. I have listened carefully to the excellent paper read by Mr. John Pattinson, and the solution which I seek seems to be more remote than ever. That gentleman stated in the course of his remarks that in the testing of soda ash, the test acid used both by Lancashire and Tyne chemists was prepared on the assumption that carbonate of soda contains  $59\frac{1}{2}$  per cent. of soda. Mr. Glover observes that though this basis is not strictly accurate, yet as it is universally observed the error will be uniform, and therefore no injustice can arise. But, sir, I assure you that the error is far from uniform. There is a difference between the results of Lancashire and Tyne chemists of from one-half to three per cent. I have divided sealed samples of soda ash into two portions, and received certificates to the following effect:—

1. 48 per cent. soda by Lancashire test.
2. 48 per cent. soda by Lancashire test.
1.  $45\frac{1}{2}$  per cent. soda by Tyne test.
2.  $46\frac{1}{2}$  per cent. soda by Tyne test.

Now, sir, in view of the method of analysis being a standard one, and the operation to be performed a very simple one, it is difficult to understand the great want of uniformity in results. No doubt the court of appeal suggested by Mr. Pattinson would be most useful. Its usefulness would also be enhanced had it the power to reprimand chemists whose results were proved conclusively to be inaccurate.

Mr. JOHN PATTINSON: I think the remarks of Mr. Davidson fully bear out the position which I took—that it is of little use merely having a method of analysis stated. We have that in the soda trade, yet



these differences between Tyne and Lancashire tests always occur. The methods for phosphoric acid, too, are well described in the books, yet differences arise between chemists using the same method. Besides stating the method of analysis, we must have courts of appeal in case of differences.

Mr. T. W. HOGG: The question affects all industries. It would be very desirable to have a fixed method of sampling pig iron, for example, because different parts of the same ingot differ in their composition.

Mr. H. R. PROCTER: The analysis of tannins at present is in such an unsettled condition that an analysis is useless unless the method is stated. I do not know whether it will be possible to get at an agreement, but I believe the German chemists have a commission at present investigating the matter, though I do not know whether they have yet come to any result. In the case of tannins, I think the way out of the difficulty will be to state the method of analysis in the contract rather than to lay down any one method.

Mr. S. DUNN (West Ham, London): I think it would be most desirable always to have the method stated, otherwise the difficulties in comparing results are very great. In acetic acid, for instance, in which I am interested, the results of different methods may vary to the extent of 8 or 10 per cent. Such a state of things wants a remedy, and the method proposed is a step likely to lead towards that.

Mr. W. W. PROCTOR: The resolution, if carried, will do much good in promoting discussion. I think the question of sampling is of quite as great importance as that of testing.

Dr. H. S. PATTINSON: The ventilation of opinion on the matter must do good, and it will, at any rate, be pointed out in what directions new or improved methods are wanted.

The resolution was then carried unanimously.

Afterwards Mr. H. R. Procter exhibited the method of determining the electro-motive force of a battery. Mr. T. W. Hogg exhibited some Sprengel specific gravity tubes, an improved gas regulator, a modification of Carmichael's apparatus for upward filtration, and an improved valve for a wash-bottle. Messrs. Mawson and Swan showed Stead's gas apparatus, gas sampler, and chromometer for determinations of carbon in iron and steel.

## Communications.

### SECTION FOR GLASGOW AND THE WEST OF SCOTLAND.

A NUMEROUSLY-attended meeting of gentlemen desiring the formation of a Glasgow and West of Scotland Section of the Society of Chemical Industry was held at the rooms of the Religious Institution, Glasgow, on the 8th of January. Professor Ferguson, of the University of Glasgow, was called to the chair. Dr. Dobbie read letters, apologising for inability to attend the meeting, from Mr. Crum, M.P., Mr. James White (of Overtown), Mr. James Mactear, Mr. Kirkpatrick, Dr. A. P. Aitken, Mr. Ivison Macadam, Dr. Falconer King, Mr. Robert Irvine, and Mr. D. Harris. Dr. Dobbie then read a requisition, signed by thirty-two members of the Society resident in the West of Scotland, asking the Council of the Society to sanction the formation of a Section having its centre at Glasgow. Mr. Stanford then moved: "That a Local Section of the Society of Chemical Industry be formed in Glasgow, to be called the Section for Glasgow and

the West of Scotland." This motion was seconded by Dr. Dobbie, and carried unanimously. On the motion of Mr. Alexander Whitelaw a committee was then appointed, consisting of Professors Ferguson, Mills, and Dittmar, Dr. Wallace, Dr. Clark, Mr. James Mactear, Mr. R. R. Tatlock, Mr. J. F. Rowan, Mr. J. B. Readman, Mr. J. Christie, Mr. T. L. Pater-son, Mr. George MacRoberts, Mr. George Beilby, Mr. J. J. Coleman (treasurer), and Dr. Dobbie (secretary). At a meeting of this committee which was subsequently held, Mr. E. C. Stanford was elected chairman of the Section, and Professor Ferguson vice-chairman. The first meeting of the new Section will be held on the 7th February. The Section already includes nearly all the leading chemists of the West of Scotland. Plenty of papers are already promised it, and it has every prospect of a most prosperous career.



### A REVIEW OF THE PRESENT METHODS OF PREPARING HYDROBROMIC ACID, AND A DESCRIPTION OF A PROCESS FOR PREPARING IT FROM BROMIDE OF ZINC AND SULPHURIC ACID.

BY AD. SOMMER, SAN FRANCISCO.

THE methods recommended for preparing hydrobromic acid may be grouped into four classes: (1) Direct combination of bromine and hydrogen. (2) Decomposition of hydrogen compounds by bromine. (3) Decomposition of the compounds of bromine with the solid metalloids by the action of water. (4) Decomposition of metallic bromides by acids.

FIRST CLASS (direct combination of bromine and hydrogen): Br and H do not combine at ordinary temperatures, not even under the influence of direct sunlight. To effect their union an elevated temperature is necessary. (a) Br and H are brought in contact with a Pt wire, kept at red heat by the electric current. (b) Br and H are passed over warmed Pt sponge. (c) Br and H are passed through a red hot platinum tube. (d) Br and H are brought in contact with a flame or the electric spark. The writer has had no personal experience of any of these processes, but according to the statements of other experimenters these methods are, although the simplest in theory, the most difficult in practice. For, on account of the corrosive action of the compounds, the apparatus can only be made of glass, porcelain, soapstone, or platinum; and to make the joints so tight that neither Br nor HBr can escape is almost impossible. The product is in all cases contaminated with Br, unless the latter be removed by passing the gaseous acid over either phosphorus or antimony. An elaborate apparatus, wherein the union of Br and H is effected by their passage through a red-hot Pt tube, has been devised by Mr. A. Harding, at Berkeley, Cal., where it has been employed at the laboratory of the University of California in preparing a fuming solution of HBr containing 65 per cent. and over of HBr. (See "New Remedies," 1882, p. 43.) Wherever a pure anhydrous acid is required these methods are preferable to all others.

SECOND CLASS (decomposition of hydrogen compounds by bromine): Bromine separates hydrogen from most hydrogen compounds, notably from hydrogen-phosphide, hydrogen-sulphide, hydrogen-iodide, ammonia, volatile oils, fixed oils, resins, alcohol, ether, etc. Under the influence of direct sunlight bromine gradually liberates oxygen from water, and forms HBr. When the mixture of bromine and water is brought in contact with substances which have an affinity for oxygen, such as sulphurous acid, arsenious acid, metals, etc., the

formation of the HBr goes on rapidly. (a) Hydrogen-sulphide is passed into a mixture of bromine and water. When little water and much bromine are employed, gaseous HBr escapes, and is conducted into water. The aqueous solution of HBr so produced is then boiled until it no longer smells of  $\text{H}_2\text{S}$ . When much water and comparatively little Br are employed, an aqueous solution of HBr is formed at once. In both cases bromide of sulphur is formed simultaneously with HBr and must be decomposed by boiling, whereby free sulphur, sulphuric acid, and an additional quantity of HBr are produced. Unless further purified the products are always more or less contaminated by one or more of the following substances: Br,  $\text{H}_2\text{S}$ ,  $\text{H}_2\text{SO}_4$ . (b) Hydrogen sulphide is passed into a solution of bromine in bisulphide of carbon covered by a layer of water. HBr is formed from the Br and  $\text{H}_2\text{S}$ , and absorbed by the water, while the liberated S is retained by the  $\text{CS}_2$ . The watery solution of HBr has to be boiled in order to free it from  $\text{H}_2\text{S}$  and  $\text{CS}_2$ , but it, too, contains  $\text{H}_2\text{SO}_4$ . (c) Bromine is passed into paraffin, which is kept at  $185^\circ\text{C}$ . One half of the Br forms bromine derivatives of the paraffin, while the other unites with the H liberated to form HBr. By continuing the heat the brominised paraffin is again split up, yielding an additional amount of HBr. (d) Bromine is mixed with sulphite or thiosulphate of sodium and a small quantity of water. Here the water is decomposed, and sulphate of sodium and HBr are formed. The HBr escapes and is conducted into water. When the evolution of HBr slackens, heat is applied. A considerable portion of HBr is, however, retained by the saline mass, and cannot be conveniently recovered. The principal objection to all of these methods is their wastefulness in material. In (a) and (b) the quantity of  $\text{H}_2\text{S}$  that has to be generated, in order to effect the conversion of the Br into HBr, is many times larger than theory would indicate, since most of it passes into the air. In methods (c) and (d), on the other hand, the yield of HBr does not correspond to the amount of Br employed, because a considerable portion of Br is retained by the residues.

**THIRD CLASS** (decomposition of compounds of bromine with solid metalloids by the action of water): Bromine unites readily with some of the solid metalloids, notably with arsenic, sulphur, antimony, and phosphorus; and these bromides, in contact with water, decompose into HBr and the hydrated oxides of the metalloids. (a) Bromide of Sulphur: A mixture of Br and S is heated with water, and thereby decomposed into HBr,  $\text{H}_2\text{S}$  and  $\text{H}_2\text{SO}_4$ . The acid is very impure and must be rectified. (b) Bromide of Antimony: Regulus antimony is digested with Br and the bromide decomposed by an excess of water. Antimonous oxide is precipitated, and HBr remains in solution. On distilling, however, traces of Sb will appear in the distillate. (c) Bromide of Phosphorus: Bromine is gradually introduced into a mixture of phosphorus and water, or phosphorus and ice, which mixture is kept cool by means of water or ice. Either common P or the red variety may be used.  $\text{PBr}_3$  is first formed, but decomposed as soon as it comes in contact with water into  $\text{H}_3\text{PO}_4$  and HBr. The HBr is finally separated by distillation. This process gives excellent results when carefully executed, but on account of the danger of explosion the greatest care must be exercised never to allow a large quantity of Br to come in contact with P. The writer has found the following method safe and profitable: Into a glass flask of a capacity of two gallons is poured one gallon of water. One pound of ordinary phosphorus is added to this, and the flask placed in a large basin of cold water. A globe-shaped separating funnel is, by means of a perforated stopper, fitted

into the flask in such a way that the end of its tube dips into the water contained therein. When everything is in order the funnel is lifted out, filled with bromine, and the stopcock opened just sufficient to allow about 60 drops of Br to be discharged in a minute. The funnel is then again transferred to the flask, and the Br allowed to drop in at the rate just stated. After about 4 lb. to 5 lb. of Br have been added, the flask is warmed till the mixture is colourless, then allowed to cool, the liquid portion poured off, the phosphorus rinsed with a little water, and the washings added to the first liquid. Through the mixed liquids is passed a slow current of  $\text{H}_2\text{S}$  until all As is precipitated as  $\text{As}_2\text{S}_3$ , and the supernatant liquid becomes perfectly clear. The solution is then filtered, the  $\text{As}_2\text{S}_3$  washed, the filtrate poured into a glass-stoppered tubulated retort, which is connected with a glass condenser and heated by an oil bath, and the HBr distilled off. The first portion of the distillate is rejected, since it smells strongly of  $\text{H}_2\text{S}$  and contains hardly any HBr. The odourless portion is preserved in glass-stoppered bottles. Although the principal products of the reaction between bromine, phosphorus, and water are, as already stated, HBr and  $\text{H}_3\text{PO}_4$ , there are also formed small quantities of phosphorus acid and arsenious acid. The former may be produced by the spontaneous oxidation of the P after all free Br has disappeared, while the latter is due to the presence of As in commercial P. By an experiment it was found that on distilling the mixture of acids just as it is decanted from the P the whole of the As will appear in the distillate. The treatment with  $\text{H}_2\text{S}$  became thereby necessary, and it accomplishes in this case the complete precipitation of the As in less than 12 hours. The quantity of the distillate necessarily varies with the amount of water in the mixture of acids. But if a strong acid is desired, it is only necessary to remove the first portions that distil over and are of a low specific gravity. The greater bulk of the HBr distils as a hydrate of the specific gravity 1.4804 at  $15^\circ\text{C}$  which contains 47.12 per cent. of HBr. At no time, but particularly not during distillation, should the pure HBr be allowed to come in contact with organic substances, such as cork, india rubber, paper, etc., since these substances induce decomposition of the acid, liberating Br. The connections between the retort and condenser should therefore be made of glass only. The phosphoric acid which remains in the retort, though not pure, is a useful by-product. When carefully executed this method gives almost the theoretical yield.

**FOURTH CLASS** (decomposition of metallic bromides by acids): A great many mineral acids, as well as several organic acids, liberate HBr from metallic bromides. Those employed in practice are, sulphuric, phosphoric, and tartaric acids. Of these, however, sulphuric acid, when of considerable strength, will liberate Br along with HBr, the  $\text{H}_2\text{SO}_4$  itself being decomposed into  $\text{SO}_2$  and  $\text{H}_2\text{O}$ . A number of experiments were made by the writer for the purpose of ascertaining the highest strength of sulphuric acid that can be employed for making HBr without giving rise to the secondary products ( $\text{SO}_2$  and Br), and it was found that so far as bromide of zinc is concerned any acid containing less than 80 per cent. of  $\text{H}_2\text{SO}_4$  can be employed without danger. (a) Decomposition of potassium bromide in water by means of tartaric acid, and decanting the aqueous acid formed from the precipitated bitartrate of potassium: The product contains some undecomposed bromide of potassium as well as bitartrate. The latter may be more completely removed by the addition of alcohol, filtering, and expelling the alcohol by evaporation. The acid produced by this method is very liable to decomposition on keeping. The writer has never seen any



such acid which did not in time turn yellow. (b) Decomposition of barium-bromide by means of dilute sulphuric acid, decanting the aqueous acid and distilling: The barium-bromide required for this purpose is made either from barium-carbonate and ammonium-bromide, or from barium-sulphide and bromine. When made by the former method it necessitates, first, the production of dry  $\text{NH}_4\text{Br}$  by adding Br to ammonia-water and evaporating to dryness. The dry salt is then treated with an equivalent quantity of  $\text{BaCO}_3$ , whereby ammonium-carbonate is formed and volatilised and  $\text{BaBr}_2$  left behind. When the latter method is employed, sulphate of barium is mixed with powdered charcoal or coal tar, placed in a covered crucible and heated to bright redness for some time. The resulting sulphide of barium is then mixed with water and Br, the mixture boiled, and the solution of  $\text{BaBr}_2$  filtered off and evaporated to dryness. In order to prepare HBr from  $\text{BaBr}_2$ , a known quantity of the latter is dissolved in water, and somewhat less than the equivalent amount of  $\text{H}_2\text{SO}_4$  added. The mixture is then allowed to settle, the clear liquid decanted, the precipitate washed with water, the washings added to the liquid, and the mixed liquids distilled. On the whole, this method is a lengthy one, although the product will be pure when due attention is paid to every particular. (c) Decomposition of potassium-bromide by phosphoric acid, and separation of the HBr by distillation: The product is pure, provided the materials were so; but the cost of the phosphoric acid prevents the general application of this method. (d) Decomposition of potassium-bromide by means of sulphuric acid, allowing the potassium-sulphate to crystallise out, separating the salt from the liquid, and distilling the latter nearly to dryness: Potassium-bromide is an expensive source of Br to the retail pharmacist when he has to buy it from the wholesale dealer. To make it himself in small quantities from a solution of potassium-hydrate and Br, evaporating, igniting with charcoal, redissolving, filtering, and evaporating is both tedious and unprofitable. Often the commercial bromide of potash contains bromate, and the acid will then contain three molecules of free Br to every molecule of bromate. Another unavoidable impurity of the distillate is sulphuric acid, from which, however, it may be freed by a rectification over barium-bromide. (e) The preparation of HBr from bromide of zinc and sulphuric acid, as originated by the writer: For the sake of convenience I shall divide this process into two parts: (1) The preparation of bromide of zinc; (2) the distillation of hydrobromic acid.

#### THE PREPARATION OF BROMIDE OF ZINC.

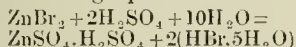
Zinc readily dissolves in a mixture of water and Br. The dissolving action, however, goes on faster when the solution has acquired a somewhat higher specific gravity than that of ordinary bromine-water, but is retarded when the solution becomes nearly saturated with bromide of zinc. In order, therefore, to utilise this peculiarity I pour the whole quantity of Br I intend to combine with Zn into a glass flask which already contains a small quantity of water, and insert a few strips of Zn, which must protrude above the surface of the Br layer. The action soon commences and is most energetic a little above the surface of the Br. When it has once fairly started, large bubbles of a colourless gas will be seen rising from the body of the Br, the nature of which gas I have not yet determined. An increase in temperature also accompanies the reaction and it is therefore well to keep the flask in a basin of cold water, and to add only a small quantity of Zn at a time. When, after repeated additions of Zn, the aqueous layer becomes

so dense that the Zn does not readily dissolve any longer, some water is added, and the additions of Zn and water repeated so long as there is any Br left. At last, an extra piece of Zn is added, and the vessel allowed to stand for some time in a warm place. This excess of Zn serves to remove most of the impurities that are generally present in commercial Zn, and particularly those which are liable to be volatilised during the distillation of the HBr, namely, arsenic, antimony, and lead. When as much of the impurities as is possible in this way have been removed, the solution is rapidly filtered and evaporated in an evaporating dish. Towards the end of the evaporation, when a pretty good heat is required to remove the last portion of water, the dish, merely protected by a piece of wire gauze, should be placed over the direct flame and the solidifying mass constantly stirred until it begins to fuse on the edges. The now granular salt, while still hot, is transferred into tightly stoppered bottles, since it is very deliquescent. Considering the ease with which  $\text{ZnBr}_2$  can be obtained in a tolerably pure state, when compared with the difficulties encountered in the production of other bromides, and, further, the advantage it has over Br in being readily handled and safely transported, it is to be hoped that it may soon become an article of commerce. If made by the bromine manufacturer, and sold cheaply, it might be made to serve as a source for Br which would not be objectionable to the transport companies.

#### THE DISTILLATION OF HYDROBROMIC ACID.

According to the statements of several investigators, aqueous hydrobromic acid, when boiled under a pressure of 760mm., has the tendency to form a stable hydrate containing five molecules of water to every molecule of HBr, which hydrate distils at  $125^\circ\text{C}$ —that is to say, when an aqueous acid is boiled under the ordinary atmospheric pressure it will either lose water, if it contains more than five molecules of water to every molecule of HBr, or HBr if the proportion is a smaller one; and it will continue to do so until there remains a liquid containing the above proportions, which will then distil over unchanged. This, however, applies, as the writer has found, only to a pure acid. In the presence of salts, and possibly, also, in the presence of other non-volatile acids, the last portions that distil are much stronger in HBr. In my experiments I therefore sought to obtain this stable hydrate, and I believe I have found a method of doing so. I take bromide of zinc 225 parts; distilled water (including the water contained in the sulphuric acid), 180 parts; sulphuric acid (calculated as  $\text{H}_2\text{SO}_4$  without water), 196 parts. The  $\text{ZnBr}_2$  is placed in a tubulated glass-stoppered retort, the neck of which is bent at an angle of about  $90^\circ$ , and drawn out to a fine point. The water is poured on the bromide, and agitated till solution is complete. Then the sulphuric acid is added (which in this case does not liberate an appreciable amount of heat), the glass stopper is tightly inserted, and fastened by a string or wire. The body of the retort is then placed in an oil-bath, the neck inclined and dipped into an empty receiver, which is standing in a larger vessel and is surrounded by water. The oil-bath is then heated rapidly, the distillation carried on as fast as possible, and the heat only lessened when excessive bumping occurs. So long as there is a considerable amount of liquid in the retort, the operation requires scarcely any attention after the heat has once been properly regulated; but when the residue in the retort is nearly dry, and the evolution of HBr slackened, close attention must be paid lest the liquid should be drawn back and fracture the retort. As soon as the liquid rises in the narrowest terminal portion

of the neck, the receiver is removed and the retort cooled and cleaned. It was found advantageous to place the neck of the retort so far into the receiver that the small orifice would be submerged in the condensed liquid, especially towards the end of the process, when a nearly anhydrous acid passes over. There is, with proper attention, no danger from the condensed liquid being sucked back, provided the orifice is made small enough, and the surface of the retort neck to which the vapours are exposed be so protected against the cooling effect of the air that no condensation, or at least only a partial one, can take place before the vapours reach the receiver, insuring thereby a constant outward pressure of the vapour from the retort. To the distillate, which contains a considerable amount of  $\text{H}_2\text{SO}_4$ , is added  $\text{BaBr}_2$ , or  $\text{Ba}(\text{OH})_2$ , or  $\text{BaCO}_3$  in excess of the  $\text{H}_2\text{SO}_4$ , and the whole again distilled to dryness. The residue from this last operation is exhausted with water, the solution evaporated to dryness, and the  $\text{BaBr}_2$  so obtained preserved for the next operation. It might appear that the rectification over  $\text{BaBr}_2$  could be dispensed with by employing an excess of  $\text{ZnBr}_2$  in the first distillation. This, however, is not the case, since by actual experiment it was found that  $\text{H}_2\text{SO}_4$  distils over even when  $\text{ZnBr}_2$  is employed in a large excess. When equivalent quantities of  $\text{H}_2\text{SO}_4$  and  $\text{ZnBr}_2$  are employed a portion of  $\text{ZnBr}_2$  equivalent to the amount of  $\text{H}_2\text{SO}_4$  that has evaporated is left undecomposed. Since, therefore,  $\text{ZnBr}_2$  is more expensive than  $\text{H}_2\text{SO}_4$ , and no advantage is derived from employing the former in excess, it is preferable to use an excess of  $\text{H}_2\text{SO}_4$ . There is still another advantage in using at least twice the theoretical amount of sulphuric acid, namely, the fluidity of the residue in the retort. Not only is the cleansing of the retort thus greatly facilitated, and its liability of being fractured lessened, but it also enables the heat to penetrate the residual mass better and expel the last portion of  $\text{HBr}$  quicker than could be the case if, on account of an insufficient amount of  $\text{H}_2\text{SO}_4$ , a hard cake of zinc sulphate were formed at the bottom of the retort. In an experiment made according to these directions I obtained 81.7 grms. of hydrobromic acid from 56.25 grm. of  $\text{ZnBr}_2$ , 63 grms. of 78 per cent. sulphuric acid, and 31 grms. of water. The specific gravity of the acid at  $15^\circ\text{C}$ . was 1.48, and its strength, determined by a standard potash solution, 47 per cent. of  $\text{HBr}$ . Hence, its molecular composition was almost exactly 1 mol.  $\text{HBr}$  to 5 mol.  $\text{H}_2\text{O}$ . It is therefore safe to express the reaction by the following equation:—



## CONVERTING SULPHIDES INTO SULPHATES BY MEANS OF NITRIC AND HYDROBROMIC ACIDS.

BY AD. SOMMER.

THE following method of converting sulphides into sulphates, although not new, I have never seen in print. Possibly it may prove of value. Sulphides, even those containing free sulphur, may be converted rapidly into sulphates by digesting them first with fuming nitric acid, and when this acid ceases to act energetically, adding repeatedly very small portions of a strong solution of hydrobromic acid. The additions of hydrobromic acid are best made through a perforated watch glass by means of a miniature funnel inserted into the opening. When the sulphides, before they are subjected to this treatment, have been reduced to an impalpable powder, their conversion into sulphates by this method can usually be accomplished in three hours.

## Journal and Patent Literature.

### II.—FUEL, GAS, AND LIGHT.

*Illumination by Gas and by Electricity in Relation to Vitiating of the Atmosphere.* M. v. Pettenkofer. Dingl. Polyt. Jour. 249, 391.

THE author has determined the percentage of carbonic anhydride and the temperature of the atmosphere of the Kgl. Residenz-Theater at Munich, under the condition of illumination both by gas and by electricity. The initial  $\text{CO}_2$  being 4 in 10,000, the following quantities were subsequently found, the house remaining empty:—

	Pit.	1st Tier.	2nd Tier.
After one half hour's illumination by gas...	5	11	14
After one hour's illumination by gas .....	6	10	20
After one hour's illumination by electricity	5	5	6

In a full house the  $\text{CO}_2$  increased to 23 in 10,000 with gas illumination and to 18 in 10,000 with electrical. The following determinations of temperature are also of interest:—

#### ILLUMINATION BY GAS.

##### Experiment 1.—Empty House.

May 2nd, 1883. Temperature in open air,  $11.8^\circ$ .

	Pit.	1st Tier.	3rd Tier.
Minimum .....	15.2	16.2	16.2
Maximum .....	16.5	19.4	25.4
Difference .....	1.3	3.2	9.2

##### Experiment 2.—Full House.

May 6th, 1883. Temperature in open air,  $11.5^\circ$ .

	Pit.	1st Tier.	3rd Tier.
Minimum .....	16.0	16.8	21.6
Maximum .....	23.2	23.6	29.0
Difference .....	6.2	6.8	7.4

#### ILLUMINATION BY ELECTRICITY.

##### Experiment 3.—Empty House.

May 29th, 1883. Temperature in open air,  $17.6^\circ$ .

	Pit.	1st Tier.	3rd Tier.
Minimum .....	16.6	17.2	17.6
Maximum .....	16.9	18.0	18.5
Difference .....	0.3	0.8	0.9

##### Experiment 4.—Full House.

June 10th, 1883. Temperature in open air,  $15^\circ$ .

	Pit.	1st Tier.	3rd Tier.
Minimum .....	17.6	18.0	18.8
Maximum .....	19.6	21.2	23.0
Difference .....	2.0	3.2	4.2

Further information on the subject is given in the *Beilage z. Ally. Zeitg.*, 1883, 177. Comp. also Fischer, *Dingl. Jour.*, 1883, 248, 375, 249, 374.—C. F. C.

*Improvements in and relating to the Manufacture of Illuminating Gas, and to the System and Apparatus Employed therefor.* H. C. Ball, Brooklyn, U.S.A. Eng. Pat. 2312, 1883.

"SLACK coal" is "exposed to the air for some time, so that a large percentage of the sulphur which exists in the coal as sulphuretted iron becomes oxidised and escapes." The coal is then washed, "as practised in making high-class coke," and it is then reduced "to an impalpable powder." This powder is then passed through a "drier and desulphuriser" (also called a "conveyor"), which is a pipe, on the outside of which is a steam-jacket, and in the inside of which is a screw. This "conveyor" is inclined, so that it may convey the powdered coal fed into it at its lower extremity into an apparatus placed at a higher level and called a "mixer." The powdered coal is dried in passing along the "conveyor," and "the sulphur which exists in it as bisulphide of iron is oxidised, and escapes as sulphurous acid," by a chimney with which the upper extremity of the "conveyor" communicates. In the "mixer," which consists of a "box within which revolves a shaft carrying a series of spirally arranged arms," the now dry powdered coal is mixed



with a certain proportion of tar. The resulting mixture is then discharged automatically into hoppers, which convey it to the retorts. The retorts are placed vertically, and the charges from the hoppers are dropped into them. The inventor claims that his process yields a gas nearly free from sulphur, of greater illuminating power than gas made in the ordinary way from the same kind of coal, that he obtains a specially hard and firm coke, that he can use cheaper forms of coal than are available for the ordinary methods of gas making, and that by his method "the labour of stoking is enormously reduced."

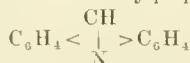
*Improvements in the Treatment and Utilisation of certain Waste Materials which have been used in Purifying Coal Gas.* J. Walker, Leeds. Eng. Pat. 2091, 1883.

A "SULPHO-ALKALINE LIQUOR" is obtained by lixiviating with water either fouled lime from gas purifiers, or the fouled mixture resulting from the purification of gas by means of the mixture of lime and coke-dust which the author of this invention proposed for that purpose in the specification of a previous patent. This liquor is then to be used "for making a size or soft starch paste or stiffening, by adding to it" various farinaceous substances, for unhairing hides or skins, and also, "in connection with other alkalis, for making common, fancy, and insecticide soaps." The solid residues remaining after the said lixiviation, mixed or not with the oxide of iron remaining after the sulphur of "spent oxide" has been burnt off therefrom, are to be employed for agricultural purposes.

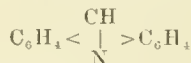
### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*On Acridine.* A. Bernthsen and F. Bender. Berl. Ber. 16, 1971.

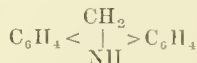
THE formula for acridine recently proposed



(Berl. Ber. 16, 1802) by the authors has led them to a new means of testing the opinions expressed (*Ann. Chem. Pharm.* 158, 265) by Gräbe and Caro regarding the constitution of soluble hydroacridine. According to the latter chemists one molecule of acridine requires one atom of hydrogen for the formation of hydroacridine, whereas the authors' experiments on hydrophenylacridine indicate a probability of two atoms of hydrogen being necessary. After unsuccessfully attempting to prepare two distinct hydroacridines, the authors ascertained by experiment how many atoms of silver one molecule of hydroacridine precipitates from a solution of silver nitrate, and, finding this number to be two, conclude that hydroacridine is formed in accordance with their theory by the addition of two atoms of hydrogen to one molecule of acridine, acridine



thus becoming hydroacridine

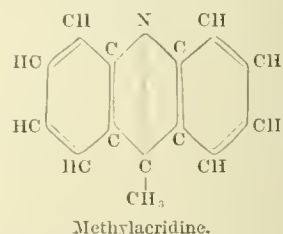
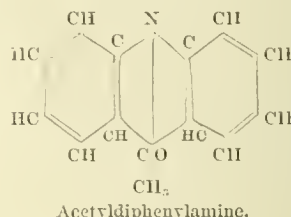
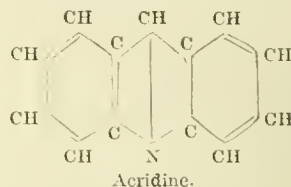


A. S.

*On Acridine.* O. Fischer. Berl. Ber. 16, 1820.

RIEDEL (*Ber.* 16, 1612) considers acridine an anthracene in which one of the two methyl groups uniting the two benzene residues is replaced by nitrogen. The author confirms this view on the following grounds. While investigating flavaniline in conjunction with Rudolph and

Besthorn a base of the formula  $\text{C}_{14}\text{H}_{11}\text{N}$  was described, which was obtained by the action of diphenylamine on acetic anhydride in presence of zinc chloride. The same base has since been prepared by Bernthsen (*Ber.* 16, 767) in a different manner. It bears great resemblance to acridine, and the author has no hesitation in assigning to it the name methylacridine. By comparing, *c.g.*, the hydrochloride of acridine with the corresponding salt of the diphenylamine base, it is found most difficult to distinguish between them, both salts having the same golden-yellow colour, crystallising in plates and exhibiting the characteristic bluish-green fluorescence in dilute aqueous solutions. According to Riedel's acridine formula the formation of methylacridine from acetyldiphenylamine is easily explained. Thus—



If this hypothesis be correct, then formodiphenylamine should lead direct to the synthesis of acridine. The author intends to investigate the synthesis of acridine from diphenylamine, formic acid, and zinc chloride.—D. B.

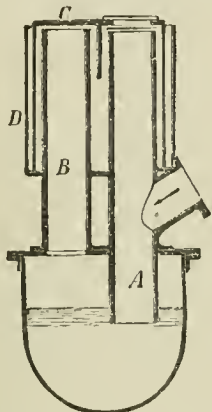
*Physiological Action of Petroleum Vapour.* Poincaré. Journ. Pharm. Chem. 7, 290.

THE author found that an atmosphere charged with petroleum vapour, such as is respired by workpeople engaged in the petroleum industry, proved fatal to guinea pigs after periods of exposure of 1-2 years; dogs and rabbits under similar treatment manifested languor and loss of appetite. The workpeople themselves complain only of an irritation of the membranes of the nose, and headache. It is nevertheless evident that in all cases precautions should be observed, with the view to prevent as much as possible respiration of these vapours by the human subject.—C. F. C.

*Pressure Reliever for Retorts, with Hydraulic Lute.* C. Pfendel, Magdeburg. Ger. Pat. 21522, May, 1882.

THE pieces of piping A and B are placed on the condenser. A dips into the luting liquid of the receiver, B enters above the same. The upper part of both pipes is surrounded by a cup D, which is also filled with a luting liquid, and into which dips the hood C, which is

divided by a partition. When the latter stands in its lowest position the communication between A and B is interrupted, so that the gas must find its way through the liquid contained in the receiver. When, however,



the hood C is raised far enough to let the partition project from the luting liquid of the cup D, the gas has an open way through the pipe B, or otherwise through the receiver.—J. G.

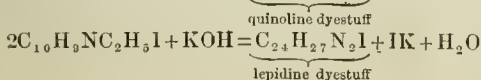
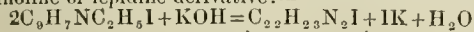
#### IV.—COLOURING MATTERS AND DYES.

*A New Method of Preparing β-Naphthylamine Sulphonic Acids.* L. Landschöf. Berl. Ber. 16, 1931.

THE conversion of β-naphthol into β-naphthylamine by the action of ammonia is not complete, and is attended with the formation of β-dinaphthylamine. The author has found, by heating at 230° to 250° the sulphonic acids of β-naphthol with a mixture of lime or soda and ammoniac, together with a little water, that conversion into the β-naphthylamine compounds is complete and unaccompanied by the formation of by-products. He has further found that this reaction is dependent only on temperature and not on pressure, hence heating in sealed tubes, which has hitherto been the procedure in this conversion, is unnecessary. The resulting mono-, di-, and tri-naphthylamine sulphonic acids submitted to the diazotization, and combined with amines or phenols, yield a series of colours which in the phenol series vary between yellow, orange, and brown, in the α-naphthol series between red and bluish violet, and in the β-naphthol series between yellow and red-orange.—A. S.

*Preparation of Colouring Matters from Coal-tar Quinoline.* W. Spalteholz. Berl. Ber. 16, 1847.

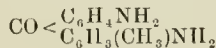
ON treating quinoline with alkyl iodides and potassium hydroxide a brown basic oil is separated, consisting, according to La Coste, of mixtures of alkylised quinolines and their ammonia bases, besides small quantities of a red or reddish-purple colouring matter which differs in shade from cyanine. The author has isolated this dye from coal tar, and subjected its ethyl derivative to a minute examination. On warming ethyl iodide with an equivalent quantity of quinoline, ethylquinolineammonium iodide is obtained, besides variable quantities of a red semi-fluid product. On recrystallising the iodide from alcohol, it is obtained in yellow monoclinic prisms. By digesting the red product with ether and recrystallising the residue from dilute alcohol, rhombic prisms of unusual magnificence were obtained, possessing a fine cantharadin green lustre. In its reactions with solvents the dye behaves like cyanine, hence it was concluded that its constitution might be analogous to cyanine, only that instead of the amyl group it would contain the ethyl group. Its formation may be illustrated by the following formulae, according as it is regarded as a quinoline or lepidine derivative:—



The numbers obtained on analysis were found to agree better with the lepidine dye. Hence it was thought that by subjecting lepidine to the preceding operation a larger yield of colouring matter might be obtained. The red colouring matter was not, however, formed, but only a small quantity of a blue dye, which was probably identical with the ethyl dyestuff obtained by Williams from the quinoline of cinchonine. In a recent communication (Ber. 16, 1082) Jacobsen and Reimer showed that quinaldine plays an important part in the formation of the red dye by the action of benzotrichloride on quinoline. The author therefore tried to determine whether quinaldine took part also in the formation of cyanine. On investigation this was found to be the case. Quinaldine ethiodide, when treated with the crystallisable (not the red semi-liquid) quinoline ethiodide, forms a dye which exhibits the same properties as the product obtained from coal-tar quinoline. This circumstance is a further proof of the identity of coal-tar quinoline with the artificial quinoline. Analyses show that the dyes obtained from crude quinoline and a mixture of pure quinoline and quinaldine are identical. The author is continuing this research with the view to ascertain how the condensation of quinoline ethiodide and quinaldine ethiodide is effected.—D. B.

*On the Decomposition of Rosaniline with Water.* C. Liebermann. Berl. Ber. 16, 1927.

THE author having characterised the non-nitrogenous substance obtained when magenta is heated to 270° C. with water as dioxybenzophenone (Berl. Ber. 5, 114, and 11, 1434), it seemed probable that the accompanying nitrogenous decomposition products might be derived from benzophenone, and not from a molecule containing 20 carbon atoms. The difference in degree of basicity of the two nitrogenous products was made use of in order to separate them from each other and to obtain them in a pure state. A crude mixture of the two, from which the dioxybenzophenone had been previously extracted, was dissolved in a small quantity of hydrochloric acid, and to the solution water was added as long as it produced a brown resinous precipitate (I.). After filtering, the filtrate was strongly saturated with NH<sub>3</sub>, whereby a large precipitate (II.) of pale red needles was produced. This was collected after 24 hours' standing, and the ammoniacal filtrate was neutralised with HCl, when a further precipitate (III.), very similar in appearance to the last, was largely produced. The filtrate from this again produced a slight precipitate (IV.) on the addition of NH<sub>3</sub>, which dissolved both in NH<sub>3</sub> and in HCl. Three-fourths of the original mixture made up the precipitates II. and III., and only these were further investigated. Precipitate II. was well washed with cold NH<sub>3</sub>, with hot NH<sub>3</sub>, with hot dilute KHO, and finally with water. The residue was dissolved in boiling alcohol, and after filtering off some undissolved impurity the filtrate was mixed with water and evaporated to crystallisation. An analysis of the recrystallised product led to the formula C<sub>14</sub>H<sub>14</sub>N<sub>2</sub>O, so that it may be considered as diaminohomobenzophenone



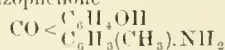
It forms colourless or pale red needles, insoluble in alkalis, but readily soluble in dilute HCl, and melting a little above 220° C. Its benzoyl derivative was prepared by heating it with six times its weight of benzoyl chloride, washing the melt with boiling water till free from benzoic acid, then with boiling alcohol, and finally crystallising the residue from glacial acetic acid. It forms nearly colourless needles, melting at 226° C. An analysis shows its formula to be that of dibenzoyldiaminohomobenzophenone



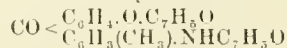
The above-mentioned precipitate III. was purified by dissolving in dilute HCl, filtering from a little resinous impurity, and reprecipitating with NH<sub>3</sub>. The precipitate thus obtained was dissolved in cold dilute KHO, reprecipitated by neutralising with acid, and, after washing with water, was crystallised from dilute



alcohol. It forms then almost colourless needles, which readily acquire a red tint. After the addition of zinc dust to its acid solution it yields colourless crystals. Its composition corresponds to the formula of an amido-oxyhomo-benzophenone



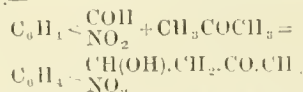
Dibenzoylamidohomobenzophenone



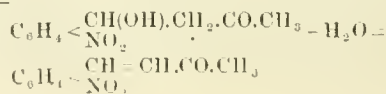
was prepared exactly as in the case of the substance above mentioned, and obtained in the form of colourless needles, m. p. 192° C. to 193° C. It seems, therefore, that when rosaniline hydrochloride is decomposed by means of water at a high temperature, the following are among the decomposition products: Dioxibenzophenone, oxy-amidohomobenzophenone, and diamidohomobenzophenone. The next higher homologue of the first and the next lower homologues of the two last are possibly also present among the products of the reaction, and the same appears to be the case with rosolic acid. Owing to the great similarity of their properties and their solubility it will, however, be very difficult to succeed in isolating them.—J. J. H.

*Paranitrobenzaldehyde and Acetone.* Ad. Baeyer and Paul Becker. *Ber. Ber.* 16, 1968.

WHEN benzaldehyde and acetone are condensed in the presence of dilute alkalis, water is eliminated according to Claisen (*Ber. Ber.* 14, 2470), and the methylketone of cinnamic acid is formed. When orthonitrobenzaldehyde is heated in the same way, there is, according to Baeyer and Drewsen (*Ber. Ber.* 15, 2856), no elimination of  $\text{H}_2\text{O}$ , but an aldole-like product—the methylketone of orthonitro  $\beta$ -phenyllactic acid—is formed, and this is only changed into orthonitrocinnamylmethylketone by the action of dehydrating substances. This difference in the behaviour of the substituted aldehyde has been explained by supposing that aldole-like bodies are formed in both cases, but that only the nitro compound possesses sufficient stability to enable it to exist under the experimental conditions imposed, whereas the non-nitrated body at once loses 1 mol.  $\text{H}_2\text{O}$ . Whether this stability might be owing to the mere presence of the  $\text{NO}_2$  group, or rather to its position in the molecule, was left an open question. The authors now answer this by studying the condensation of paranitrobenzaldehyde and acetone, and find that the nitro group in the para position acts exactly the same as if it were in the ortho position. *Paranitro- $\beta$ -Phenyllactic Acid Methylketone:* One part paranitrobenzaldehyde is dissolved in six to eight parts acetone, the solution is cooled with a freezing mixture, and a 1 per cent. solution of  $\text{NaHO}$  is slowly added with continual stirring until the liquid is slightly alkaline. Should any of the aldehyde separate by reason of the low temperature, the mixing vessel is removed from the freezing mixture till it redissolves. After neutralising the product with  $\text{HCl}$ , excess of acetone is distilled off, and the remaining oil and  $\text{NaCl}$  solution is poured on a large watch-glass. The oil coagulates, and the pressed mass is crystallised from ether. The following equation shows the formation of the condensation product:—



It melts at 58° C., and dissolves readily in ether, alcohol, benzene, and glacial acetic acid, but is insoluble in ligroin and cold water. If boiled with acetic anhydride, acids, or even water, it is changed into paranitrocinnamylmethylketone (paranitrobenzylideneacetone), which melts at 110° C., and is identical with the product obtained by direct nitration of benzylideneacetone. Evidently, therefore, elimination of  $\text{H}_2\text{O}$  readily takes place in the para series:—



Mild oxidising agents, e.g., glacial acetic acid and  $\text{K}_2\text{Cr}_2\text{O}_7$ , change the condensation product into paranitrobenzoic acid, m.p. 237° C. Special interest is attached to the behaviour of the condensation product towards alkalis, since the latter change the ortho compound into indigo. *Paranitrocinnamylmethylketone:* To a dilute solution of the above-described lactic-acid-ketone in hot water, a 30 per cent. solution of  $\text{KHO}$  is added drop by drop, very slowly and with continual stirring, until a crystalline yellow precipitate is produced. If this is collected and crystallised from acetone it forms lustrous sulphur-yellow scales, little soluble in alcohol and acetone, not at all in ligroin and water, but readily soluble in glacial acetic acid. Its m.p. is 254° C. An analysis proves it to be paranitrocinnamylmethylketone. When oxidised by means of glacial acetic acid and  $\text{K}_2\text{Cr}_2\text{O}_7$ , it yields paranitrobenzoic acid, which shows that the nitro group has taken no part in the reaction. A participation, too, of the benzene nucleus is very improbable, because of its great stability in the nitrated condition, so that there only remains the supposition that the  $\text{H}_2\text{O}$  has been eliminated from a side chain, and accompanied by a condensation between the side chains. This explains the total difference of the substance from paranitrocinnamylketone and its great insolubility, but whether the condensation takes place between two or three molecules is not determined. The result, then, of the study of the action of alkalis on paranitro- $\beta$ -phenyllactic acid ketone is as follows: They effect the elimination of  $\text{H}_2\text{O}$  as well as a molecular condensation, whereas the action of acetic anhydride and of boiling water is simply that of water elimination. The products have no similarity with indigo, which is formed by the action of alkalis on the corresponding ortho compound, because the nitro group apparently remains intact when it is in the para position, whereas when in the ortho position its oxygen is transferred to the side chain, and acetic acid being eliminated, the atomic arrangement peculiar to indigo is brought about. The first stages of the reaction seem to be the same in both cases, however, and the above study shows that the initial move in the direction of the formation of indigo from nitro substitution products is the elimination of water from the side chain, whereby a complex atomic group is produced, which, in order to preserve equilibrium, depends not on the side chain of its own or of another molecule, but on the more promising ortho-nitro group.—J. J. H.

*Contribution to our Knowledge of the Colouring Matter obtained from Dimethylaniline and Chloranil.* H. Wichelhaus. *Ber. Ber.* 16, 2005.

THE author has at length succeeded in obtaining the colour base produced by the action of chloranil on dimethylaniline, in a pure state (*Ber. Ber.* 14, 1942). The base must be only incompletely precipitated from the salt solution in order to retain all excess of dimethylaniline in solution; it is then washed with water, dried, rubbed up with  $\text{NaHO}$  in order to remove traces of acid, washed, dried, and crystallised from ether. It forms small colourless prisms, insoluble in water, and little soluble in alcohol. Its melting point is 190° C. Its composition ( $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}$ ) is the same as that of the base of ordinary methyl-violet, but the properties of the latter are somewhat different. A fresh examination, therefore, of the base of methyl-violet seemed desirable, and the result has been to show that although its formula corresponds to the one just given, it is nevertheless a mixture containing about 27 per cent. of the above-mentioned crystalline base. A solution of methyl-violet, specially prepared from pure dimethylaniline, was precipitated with alkali, and the base thus obtained formed a reddish-brown readily-fusible powder, having the composition  $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}$ . It was repeatedly dissolved in dilute  $\text{H}_2\text{SO}_4$ , and as often precipitated with alkali without showing any change in appearance. It was then boiled with several quantities of ligroin, as long as anything dissolved, and in this way 30 grms. could be separated into 8 grms. of a crystalline body, and 22 grms. of a reddish-brown powder. Both products are very similar to each other, and dye wool and silk violet. The first

base may be crystallised from ether or from hot alcohol. Its melting point is  $190^{\circ}\text{C}$ ., and it possesses the same properties and the same crystalline form as the base obtained by means of chloranil. The second base, which amounts to over 70 per cent. of the base of methyl-violet, is a reddish-brown powder, insoluble in ether, and with melting point  $130^{\circ}\text{C}$ . When reduced by means of Sn and HCl, both bases yield different crystalline products. The first gives lustrous leaflets (melting point  $176^{\circ}$ ), which soon acquire a violet colour. They are identical with the reduction product of the chloranil derivative. The second base on reduction yields silver-gray prisms (melting point  $155^{\circ}$ ), which retain their gray tint for a lengthened period. It would appear, therefore, that the base derived from chloranil should have the formula  $\text{C}_{24}\text{H}_{30}\text{N}_3$ , and not  $\text{C}_{16}\text{H}_{20}\text{N}_2$  as was first proposed, and that it is probably an isomer of what forms the largest proportion of the base of methyl-violet.—J. J. H.

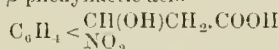
*On the Colouring Matter of Cochineal.* By H. Fürth. Berl. Ber. 16, 2169.

LIEBERMANN and Van Dorp (*Ann. Chem. Pharm.* 163, 97) obtained rubicoccin ( $\text{C}_{16}\text{H}_{10}\text{O}_6$ ) by the action of  $\text{H}_2\text{SO}_4$  on carmine, and by distilling rubicoccin with zinc powder they obtained a minute quantity of a hydrocarbon ( $\text{C}_{16}\text{H}_{12}$ ). The author obtained this same body by reducing in a similar way either coccinin (*Ann. Chem. Pharm.* 141, 329) or carmine itself, but in both cases the amount obtained was very small. When coccinin, mixed with many times its bulk of zinc powder, is heated in a tube, white vapours are first given off, which partly condense in the form of greenish-coloured leaflets; these were mixed with the yellow distillation product, also obtained in the condenser, and purified by repeated crystallising from ether, alcohol, and benzene, and finally by sublimation. The product (m.p.  $186^{\circ}$  to  $187^{\circ}$ ) is identical with that obtained from rubicoccin. If coccinin is heated with acetylchloride to  $100^{\circ}\text{C}$ . in a sealed tube, an acetyl compound of coccinin is produced, which separates from a hot alcoholic solution on cooling in the form of yellow crystals. It is insoluble in water, but readily soluble in alcohol and in glacial acetic acid. If the formula of the above-mentioned hydrocarbon is accepted as  $\text{C}_{16}\text{H}_{12}$ , the formula of coccinin is found to be  $\text{C}_{16}\text{H}_{14}\text{O}_6$ , and that of its acetyl derivative  $\text{C}_{16}\text{H}_{10}\text{O}_2(\text{C}_2\text{H}_3\text{O})_4$ . Coccinin would therefore appear to be the hydrate of a quinone of the hydrocarbon in question, and containing four hydroxyl groups. The hydrocarbon is also obtained from carmine, by simply heating the latter in a tube with zinc powder. The distillation product is dissolved in ether, the solution is shaken with KHO, then with HCl, and thus purified is allowed to crystallise. Its m.p. is  $186^{\circ}$ . It is identical with that obtained from rubicoccin by Liebermann and Van Dorp, and with that obtained from coccinin.—J. J. H.

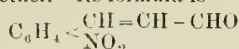
*Action of Orthonitrobenzaldehyde on Aldehyde.* Ad. Baeyer and V. Diewsen. Berl. Ber. 16, 2205.

IN a former paper (*Berl. Ber.* 15, 2856) the authors mention that by the action of a little  $\text{BaH}_2\text{O}_2$  water, on a solution of orthonitrobenzaldehyde in aldehyde, a crystalline body (m.p.  $120^{\circ}$ ) is produced. In order to prepare it, a 2 per cent. solution of NaHO is carefully added drop by drop, and with constant stirring, to an ice-cold solution of orthonitrobenzaldehyde in freshly-made aldehyde, until the alkaline reaction is persistent for at least five minutes. The liquid is then poured into a basin, excess of aldehyde is driven off by a current of air, and the crystalline product remaining is at once placed on an earthenware absorbent plate, and finally recrystallised from ether. It forms large colourless monoclinic prisms. It is readily soluble in alcohol, chloroform, etc. It melts at  $125^{\circ}\text{C}$ ., and gives off aldehyde vapour. An analysis gives numbers which correspond to a compound of orthonitrophenylactic-aldehyde with aldehyde ( $\text{C}_9\text{H}_7\text{NO}_4 + \text{C}_2\text{H}_4\text{O}$ ). Its aqueous solution gives off aldehyde even at a temperature of  $40^{\circ}$  to  $50^{\circ}\text{C}$ ., but on leading a current of air through such a solution it was not possible to obtain the free orthonitrophenylactic-

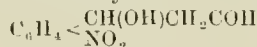
aldehyde in a crystallisable state. Both the free and the aldehyde compound, when acted upon by alkalis, give indigo. The free substance combines with a solution of a bisulphite, and reddens magenta sulphite. *Orthonitro- $\beta$ -Phenylactic Acid:* A solution of the condensation product in dilute alcohol is heated on a water bath with an excess of freshly-precipitated  $\text{Ag}_2\text{O}$  until a sample no longer yields indigo on the addition of alkali. The liquid is then boiled with HCl, filtered from AgCl, and extracted with ether. On evaporating the ethereal extract a crystalline mass possessing acid properties remains behind. It is purified by preparing its barium salt and decomposing this with HCl. Crystallised from water it forms short prisms (m.p.  $126^{\circ}\text{C}$ .). It dissolves readily in water, alcohol, and ether, and is proved by analysis to be orthonitro- $\beta$ -phenylactic acid



When heated to  $190^{\circ}\text{C}$ . with dilute  $\text{H}_2\text{SO}_4$ , it gives orthonitrocinnamic acid. It is identical with an acid obtained recently by Einhorn from the addition product of HBr with orthonitrocinnamic acid. When heated with concentrated  $\text{H}_2\text{SO}_4$  it gives a blue solution containing apparently indoin. *Orthonitrocinnamic aldehyde:* This is produced by boiling the condensation product with acetic anhydride for about one hour until a sample on addition of NaHO no longer yields indigo. The excess of acetic anhydride is then driven off by evaporating, and the residual hot solution is decolourised with animal charcoal. The filtered liquid on cooling yields colourless needles which are recrystallised from ether. The pure substance melts at  $127^{\circ}\text{C}$ . It dissolves readily in boiling water and in chloroform, but only little in cold water, alcohol, and ether. Its formula is



It possesses all the properties of an aldehyde, forming a crystalline compound with sodium bisulphite, reducing an ammoniacal silver solution with formation of orthonitrocinnamic acid, and when reduced itself yielding quinolin. The above shows, therefore, that the condensation of orthonitrobenzaldehyde with aldehyde takes place in the same sense as that with acetone when orthonitro- $\beta$ -phenylactic aldehyde



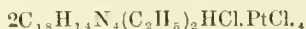
is formed, which, however, forms a still more unstable union with acetaldehyde.—J. J. H.

*New Coal-tar Colouring Matters.* Dingl. Polyt. Jour. 249, 383. (a) *New Yellow, Orange, and Brown Colouring Matters.* E. Nörling and E. v. Salis, Mayenfeld. Ger. Pat. 22268, 1882. (b) *Flavulin.* E. Besthorn and O. Fischer. Dingl. Polyt. Jour. 249-385 (Comp. ibid. 247-48). (c) *Colouring Matters of the Saffranin Group.* R. Nietzki. Dingl. Polyt. Jour. 249, 386. (d) *Observations on the Saffranin Group.* R. Bindschedler.

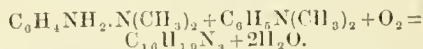
(a) THESE are the  $\text{SO}_3\text{H}$  derivatives of nitrated secondary, and tertiary aromatic amines, or of tertiary amines containing two aromatic and one fatty radicle. They are obtained either by sulphonating the corresponding nitramines, or by the action of halogen-nitro compounds upon aromatic amidosulphonic acids. The following halogen-nitro compounds have been employed: Chlorodinitrobenzene, chlorotritrobenzene, or the chlorodinitrotoluene, parachlorodinitrotoluene, chlorodinitronaphthalene, chlorotritronaphthalene, chlorotetranitronaphthalene, in reaction with the following amines: aniline, ortho- and para-toluidine, the xylidines, methyl-ethyl aniline and homologues, diphenylamine, phenyltolylamine and ditolylamine, dinaphthylamine, amidoazobenzene, its homologues and substitution derivatives, etc. The resulting nitramines are converted by sulphonation into soluble bodies, which are the colouring matters in question, or such soluble bodies may be directly produced by using in place of the amine its  $\text{SO}_3\text{H}$  or  $\text{COOH}$  derivative. As an example, trinitrodiphenylaminesulphonic acid is described by the authors. It



may be directly obtained by heating together, in a flask attached to an upright condenser, 3 parts (1 molecule) sulphuric acid and 3 parts (1 molecule) picrylchloride with 2.5 parts sodium acetate in concentrated aqueous solution. The boiling is continued until the picrylchloride completely disappears; or the reaction may be more rapidly determined by heating under pressure at 120° to 150°. The following compounds are also mentioned as of special technical importance; the products of the action of picrylchloride, chlorodinitrobenzene, and chlorotetra-nitronaphthalene, upon amidazobenzene mono- and di-sulphonic acids, and phenylamidazobenzene sulphonic acid. The reaction is of the same character as that above described. The proportions of the reagents are 35 parts amidazobenzene disulphonic acid or of phenylamidazobenzene sulphonic acid, 40 to 45 parts sodium acetate in concentrated aqueous solution, 20 parts chlorodinitrobenzene, or 25 parts picrylchloride or 35 parts chlorotetra-nitronaphthalene. (b) When flavolin ( $C_{16}H_{13}N$ ) is nitrated with fuming  $HNO_3$  it yields a nitro body, which on reduction is converted into flavaniline or monamido-flavolin,  $C_{16}H_{12}NH_2N$ . Flavaniline is also formed by the action of acetyl chloride upon aniline sulphate at 100°, or upon acetanilid in presence of zinc chloride. It is evident, therefore, that valuable colouring matters are obtainable from complicated quinoline derivatives; and it is probable that such may be obtained from the high-boiling quinoline bases of coal tar, e.g., acridin. (c) Witt obtained (this Journal, 1882, p. 225) what he regarded as the first member of the safranin series of colouring matters by the oxidation of a mixture of paraphenylenediamine (1 molecule) and aniline (2 molecules). The author has extended Witt's observations, using as his oxidising agent potassium bichromate in hot solution. He obtained similar compounds by using, in place of the aniline, ortho- or meta-toluidine, a mixture of either of these monamines (1 molecule) with paratoluidine (1 molecule), or a mixture of mono- or di-methylaniline (1 molecule) and any primary monamine (1 molecule), but not with secondary or tertiary bases, nor with paratoluidine alone. Similarly, in place of paraphenylenediamine, certain of its substituted derivatives may be used. Those, however, which contain an alcohol radicle in each  $NH_2$  group do not react. Diethylparaphenylenediamine oxidised in presence of aniline (2 molecules) yields diethylsafranin, a magenta-red colouring matter. Its platinumchloride, dried at 100°, has the composition

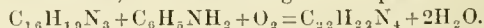


A mixture of paraphenylenediamine with aniline (1 molecule) and diethylaniline (2 molecules) yields on oxidation a body isomeric with the above. Both bodies yield green salts with sulphuric acid, but they neither form a green diazo-compound. *Tetrathylsafranin* is formed from dimethylparaphenylenediamine, diethyl-aniline, and aniline. It differs from the preceding compounds in being much more violet in shade. It is observed in all cases that the formation of the safranines is preceded by that of blue or green colouring matters, representing the intermediate stages of oxidation. With regard to the constitution of the safranines it is now proved that they contain two amido-residues, united by an N-atom, which occupies the para-position in regard to the N-atoms of the amido groups, the simplest safranin containing the group  $H_2N.C_6H_4.N.C_6H_4.NH_2$ . Its analogies with rosaniline are evident. When phenosafranin is heated with concentrated HCl at 170° it yields a product which has the closest resemblance to aniline black. (d) If dimethylparaphenylenediamine (1 molecule) and diethylaniline (1 molecule) be treated with potassium dichromate in aqueous solution at 30° C., and in presence of zinc chloride, a separation of copper-coloured crystals,  $2C_{16}H_{12}N_2HCl.ZnCl_2$ , occurs in a few minutes. The new colouring matter, dimethylphenylene-green, is formed according to the equation—

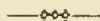


It yields on reduction a corresponding leuco base. A corresponding ethyl derivative could not be obtained. If the above-mentioned zinc chloride compound be oxidised

in presence of aniline acetate it yields a beautiful violet colouring matter, which appears to be tetramethylphenylene safranin, formed according to the equation—

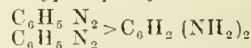


Similar results are obtained by oxidising the dimethylphenylene-green in presence of para- and ortho-toluidin, xylinin, etc. Dimethylparaphenylenediamine and aniline (2 molecules) yield on oxidation dimethylphenylene safranin ( $C_{26}H_{18}N_4$ ). Safranines are decolourised on reduction. A solution of pure phenylene safranin reduced in an atmosphere of hydrogen gas by means of a titrated solution of ferrous ammonium sulphate, was found in passing into the leuco compound to combine with 4H. The general formula for the safranines is  $C_nH_{2n-22}N_4$ .—C. F. C.



*Process for Preparing Brown Azo-Colours from Chrysoidines and Diazo Compounds.* Actiengesellschaft für Anilinfabrikation, Berlin. Ger. Pat. 22714, Nov., 1883.

THE chrysoidines (e.g., diamidazobenzene hydrochloride) or their sulphonic or carbonic acids can combine with a second molecule of diazotised base or amido acid, forming colours of the type of phenylenediaminediazobenzene



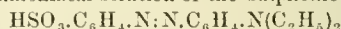
The ordinary chrysoidine, e.g., yields with diazobenzene-chloride a brown colour. These colours can be converted into sulphonic acids, or the sulphonic acids of the components can be taken as the first materials from which to work.—J. G.

*Method for the Manufacture of Yellow Colouring Matters from Pyridine and Quinoline Bases.* Emil Jacobsen, Berlin. Ger. Pat. 23188, Nov. 4, 1882.

By heating phthalic anhydride with zinc chloride and pyridine or quinoline bases, to a temperature of 200° to 250°, yellow colouring matters are formed, which can be converted into soluble sulphonic acids. Similar dyes are obtained by using quinaldine in the place of pyridine or quinoline.—F. M.

*Method for the Preparation of a Blue Colouring Matter from Diethylaniline-azobenzene-para-sulphonic Acid.* Lembach and Schleicher, Biebrich-on-the-Rhine. Ger. Pat. 23278, Dec. 3, 1882.

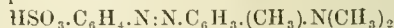
To the ammoniacal solution of the sulphonic acid—



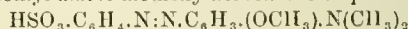
obtained from diethylaniline and para-diazo-benzene sulphonic acid, zinc-dust is added until the liquor is decolourised. The solution of the hydrazo-compound thus formed is acidulated and saturated with sulphuretted hydrogen. On adding ferric chloride a deep blue solution is produced, from which the colouring matter may be precipitated by common salt.—F. M.

*Method for the Preparation of Blue Colouring Matters.* Otto Mühlhanser, Colour Works, Griesheim-on-the-Maine. Ger. Pat. 23291, Jan. 5, 1883.

LAUTH'S reaction (treatment with sulphuretted hydrogen, and subsequent oxidation) is applied to the sulphonic acids of the amidoazo derivatives of substituted toluene and anisol. For instance, dimethyl-amido-methyl-azobenzene-sulphonic acid—



or dimethyl-amido-methoxy-azobenzene-sulphonic acid—



F. M.

*A New Blue Colour to be used as a Substitute for Indigo for Dyeing Cottons, Wool, and Silk.* William Heather Spence. A communication from Auguste François Chesnais, of Ronen. Eng. Pat. 1589, March 29, 1883.

THE inventor claims the use of a solution of an induline salt mixed with nitrosodimethylaniline blue (sulpho-conjugated) or methylaniline violet, etc., the application of

this mixed colour to the fabric, and its oxidation thereon by means of potassium dichromate. In order to prepare the solution the induline base is converted into its sulphate, acetate, or hydrochloride, and the salt dissolved in alcohol in the proportion of 120grms. to the litre. The methylaniline violet or nitrosodimethylaniline blue (sulpho-conjugated) is then added, and the liquid is said to be "fit for commerce." The dye is applied to the fabric by "tannate of tin," and then oxidised by passing through a chromate bath.—R. M.

*Improvements in Red Dyestuffs suitable for Dyeing and Printing.* Sydney Pitt. A communication from the Verein Chemischen Fabriken, Mannheim. Eng. Pat. 2237, May 2, 1883.

THE new dyestuffs are azo compounds derived from alpha-naphthol instead of beta-naphthol. The sulphonic acids of alpha-naphthol are combined with the diazo derivatives of aniline, toluidine (ortho and para), xyldine, and other homologous bases, amidoazobenzene and its homologues, the naphthylamines, etc. Two methods are given for the manufacture of the sulphonic acids, the first consisting in the sulphonation of the naphthol by means of ordinary or fuming sulphuric acid at a low temperature, and the treatment of the mixed sodium salts thus obtained with boiling alcohol, which leaves the useless isomeride insoluble. In the second method (which is preferred) three modifications of alpha-naphthol sulphonic acid are claimed, these being derived from alpha-naphthylamine by the following processes: (1) Naphthylamine is mixed with the theoretical quantity of sulphuric acid and the mixture heated to 150° to 180° C. till soluble in alkali. (2) Naphthylamine is dissolved in from four to five times its weight of ordinary sulphuric acid, and heated to 100° to 120° C. till completely sulphonated. (3) Dry naphthylamine hydrochloride is stirred into cold fuming sulphuric acid containing 20 to 22 per cent. of free anhydride. These naphthylamine sulphonic acids are converted into the corresponding naphthol sulphonic acids by the usual diazo reaction, viz., by treatment with a nitrite and then boiling with water or dilute acid. The three sulphonic acids are stated to be isomeric, and all applicable for the production of red dyes by combination with the diazo compounds specified.—R. M.

*Improvements in the Manufacture of Dyes.* Gustav Adolph Bang. A communication from Gustav Adolph Dahl, Ernst Dahl, and Rudolph Dahl, Dahl and Co., Barmen. Eng. Pat. 2296, May 5, 1883.

THIS invention claims the production of azo dyes by the action of diazobenzene and its homologues, diazoanisole, diazonaphthalene, and diazo-azo compounds upon what is described as the "monosulphate of alpha-naphthaline derived from naphthaline" (alpha-naphthol-monosulphonic acid?). The dyes are said to be yellow-red, scarlet, and blue-red.—R. M.

*Improvements in Converting Anthraquinone into a Monosulpho Acid of Anthraquinone, also applicable for treating other organic compounds.* Ivan Levinstein. Eng. Pat. 2299, May 7, 1883. Provisional protection only.

THE improvement consists in heating the quinone with fuming sulphuric acid in a closed enamel pan provided with an agitator having a hollow shaft, through which the contents are discharged by air pressure when the operation is complete.—R. M.

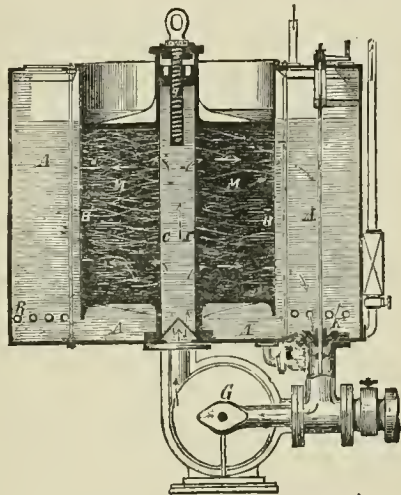
*Improvements in the Manufacture of Alpha- and Beta-Naphthol.* Ivan Levinstein. Eng. Pat. 2300, May 7, 1883.

IN making the naphthols by the usual method of fusing the sodium salts of the naphthalene-sulphonic acids with caustic alkali, the inventor allows the melt to separate into two layers, the upper of which consists of sodium naphtholate and the lower of sodium sulphite, hydroxide, and impurities. The lower layer is utilised for the preparation of the sodium salts of the naphthalene-sulphonic acids. The sodium naphtholate which forms the upper layer is removed and decomposed by acid in the usual way.—R. M.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

*Method and Apparatus for Treating (Washing, Dyeing, etc.) Textile Fibres, Yarn, and all kinds of Cloth.* Otto Obermaier, Lambrecht, Pfalz. Ger. Pat. 23117. Dec. 6, 1882.

THE apparatus consists of a vessel with a charging space M, in which the textile stuff is compressed, and held compressed by a screw piston. This vessel is composed of two concentric cylinders BC, with perforated sides and closed bottoms, the inner cylinder being set upon a pipe which is connected to a pump. The vessel stands in an outer vessel A, provided with a heating arrangement R, and connected by a valve with the pump G,



and it is so arranged that the liquid, which is forced at a high pressure by the pump G into the perforated cylinder C, penetrates the textile stuff M, and passes through the perforations from B to A, where it is warmed again, and then passes through the valve K back into the pump, in order to begin the round again. The vessel for holding the textile stuff can be made to rotate. For filtering purposes, and in order to keep back pieces of fibre, and to prevent the textile stuff from coming into contact with the metal surfaces of the apparatus, a filter is placed in the smaller cylinder, and the perforated sides of the charging space are covered with cloth. Several of these apparatus may be united to form a battery, in which case the single basins A are arranged together and connected with one another.—W. M.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Bistres for Dyeing and Printing.* Dingl. Polyt. Journ. 249, 35, 1883. From the Moniteur Scientifique, September, 1882.

ACCORDING to Naquet, a good solution for producing a bistre on cotton fabrics is composed as follows: Bismuth, 100 parts; nitric acid, 280 parts; tartaric acid, 75 parts; ammonia, 0.06 part; sodium thiosulphate, 75 parts; water, 2,000 parts. Fabrics saturated with this solution, when dried at 25° to 30°, gradually develop a clear chestnut brown, whose intensity reaches a maximum after 24 hours. By steaming the dried saturated fabrics there is produced a darker chocolate catechu, which is much intensified by treatment with a weak solution of sodium thiosulphate. In this case, owing to the complete conversion of all the bismuth into sulphide, the fabric possesses a dull yellowish tint and unpleasant metallic lustre. The above solution does not deteriorate on keeping, even in contact with the atmosphere. Upon the constitution and production of manganese-bistre, recent experiments by Balanche throw some light. It is found that no reaction takes place between manganous



chloride and potassium bichromate, but that if the latter be substituted by the monochromate an abundant bistre-coloured precipitate falls. By printing, therefore, with the following mixture—180grms. potassium dichromate, 850grms. water, 150grms. wheat-starch paste, 210grms. manganous chloride, 210grms. sodium acetate liquor of 16.5° B., and steaming, a dark well-fixed bistre is developed which withstands washing and soaping, and does not injure the fabric. The formation of this colour is here due to the decomposition of the sodium acetate, and consequent conversion of the potassium bichromate into the monochromate. The constitution of this bistre is possibly  $MnCrO_4 \cdot MnO_2 \cdot H_2O$ , and is at present the subject of further investigation.—W. D. B.

*An Improved Process of Turkey-Red Dyeing for Cotton Yarns and Cloth.* John William Hoffmann. A communication from Carl Seeber, Schopfheim. Eng. Pat. 2289, May 5, 1883. Provisional protection only. THE yarn is first treated with a boiling solution of soda ash and resin, then with an oil mordant composed of Turkey-red oil, "stannate of soda," and ammonia liquor. An alumina mordant is then applied, and finally a chalk bath. The inventor claims a saving of time and economy of cost.—R. M.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Preparation of Ammonium Sulphocyanide.* J. Schulze. Journ. f. Prakt. Chem. 27, 578.

THE author recommends for this purpose the action of ammonia upon carbon disulphide in alcoholic solution, as already proposed by Millon. From 600grms. alcohol (95 per cent.), 800grms. ammonia solution (sp. gr. 0.912), and 350grms. carbon disulphide, the author obtained 250grms. of dry ammonium sulphocyanide.—C. F. C.

*On Aluminium Sulphocyanide free from Iron.* Dr. Gottlieb Stein. Dingl. Polyt. Journ. 250, 36.

SINCE the introduction by Stork and Strobel (*Dingl.* 241, 464) of aluminium sulphocyanide, in the place of aluminium acetate for making alizarin steam-reds, the author states that it has become a point of importance in buying aluminium sulphocyanide to obtain it as free from iron as possible, since preparations containing iron produce duller and bluer reds than those which have been purified from iron. All commercial samples of aluminium sulphocyanide show the presence of iron when tested with yellow prussiate of potash, and it is present in largest quantity when ammonium sulphocyanide obtained from gas-liquor has been used to produce the aluminium compound. The iron exists in the preparation chiefly in the form of ferric sulphocyanide,  $Fe_2(CNS)_6$ , and since this compound dissolves in ether, as is well known, forming a red solution, while the aluminium compound, on the other hand, is not very soluble, the author has tried the process of extraction by ether on the scale of a laboratory experiment, and finds that by treating 1 kilog. of aluminium sulphocyanide, of sp. gr. 2.0° B. (containing 25 per cent. sulphocyanide), with three successive portions of 660grms. each of ether, he obtains a preparation of aluminium sulphocyanide which shows but a faint trace of iron when tested with potassium ferrocyanide. The ether which mixes to some extent with the aluminium sulphocyanide reduces the specific gravity of the solution after the third extraction to 1.2° B., and to prevent the separation of sulphur which takes place if the ether is left in the solution it must be rapidly distilled or evaporated off. This process of extraction can be used also as an analytical method for determining the quantity of iron in a sample of aluminium sulphocyanide, by evaporating the ether solution to dryness, and estimating the iron in the residue by any of the well-known methods.—H.S.P.

*Improvements in Concentrating Sulphuric Acid, and in Apparatus therefor.* W. P. Thompson, Liverpool. Communicated by W. West, Denver, U.S.A. Eng. Pat. 1651, 1883.

SULPHURIC acid of from 48° to 50° B. is evaporated in

a lead pan up to 60° B. It is then fed into a cast-iron retort interiorly "lined or enamelled with porcelain or other vitreous substance." In this it is evaporated to a density of 66° B. Arrangements are described by which the operation can be conducted continuously.

*Improvements in the Mode of Concentrating Sulphuric Acid and in Apparatus for the same.* S. Bowen Bowen, Llanelly. Eng. Pat. 2035, 1883.

THE author of this invention uses "a series of retorts made of glass or any other suitable material, with inlet and outlet passages in the sides or ends of each retort sufficiently large to allow the volume of weak vapour to pass from the lowest retort through the series, and also serving to conduct the acids from the highest to the lowest retort, by means of glass tubes or adapters connected to each retort, the weak vapour ultimately passing through a small condenser to the chimney. By the vapour passing through the series, the more dense vapour of the lower retorts becomes condensed as it passes through the space of the adjoining retorts, so that when it leaves the last retort it is in a very weak state." The retorts should be arranged at successive elevations, "so as to allow the acid to run from the highest retort to the lowest. . . . The retorts are placed in chamber spaces, heated by fires underneath. Hot acid of specific gravity 1.750" is fed into the topmost retort of the series, and concentrated acid flows off from the lowest one. An arrangement is described for automatically regulating the supply of acid to the topmost retort.

*Improvements in the Manufacture of Oxide of Strontium and Hyposulphite of the Alkalis.* W. Moody, West Ham. Eng. Pat. 2259, 1883.

HAVING obtained strontium sulphide by deoxidising strontium sulphate by furnacing it in admixture with coal, the author of this invention mixes the said sulphide, while still hot, with water, "to form a thin pasty mass." He then boils this mass with a quantity of caustic alkali "sufficient to combine with the sulphur of the sulphide." On cooling, strontium hydrate crystallises out. The mother-liquor consists of a solution of sulphide of sodium or of potassium, according to the alkali which has been employed. This may either be simply concentrated by evaporation until hydrated sulphide will crystallise from it on cooling, or it may be treated by sulphurous acid, for the conversion of its sulphide into hyposulphite with precipitation of free sulphur.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Improvements in the Manufacture of Sulphate of Lime or Plaster-of-Paris.* J. H. Johnson, London. Communicated by P. G. Jourmet, Paris. Eng. Pat. 2566, 1883.

THE author of this invention makes slaked lime into a paste with dilute sulphuric acid, partially dries that paste, and then calcines it, thus obtaining either "anhydrous plaster or a mixture of anhydrous plaster and lime," according to the proportion of sulphuric acid which has been used. By varying the proportions of sulphuric acid employed he obtains "a large variety of products of similar nature, but differing in degree of hardness and in the time they require for setting."

*Improvements in the Manufacture of Artificial Stone and Cement.* E. S. Shepherd, Paddington, and J. L. Aspinwall, Nantlle Vale, Carnarvon. Eng. Pat. 2352, 1883.

THE author of this invention proposes to employ in the manufacture of artificial stone and cement the debris of slate quarries.

*A New or Improved Combination of Materials for making Cements, capable also of being used for other useful and ornamental purposes.* T. Smith, Sunbury. Eng. Pat. 2199, 1883.

THE scrapings of macadamised roads are washed to remove from them "all light or superfluous matters," and are then "thoroughly baked," and afterwards finely ground. The product is then mixed with dry powdered chalk and coke-dust, in the proportions of 43 parts of the said product to 20 parts of chalk and 37 parts of coke-dust, and the mixture so obtained is then incorporated with molten sulphur. Sand may be used instead of the road scrapings, and for some applications of the composition, as for jointing pipes, pitch or bitumen may be substituted for a part of the sulphur.

*Improvements in the Manufacture of Artificial Stone for various Purposes and Material applicable as a Substitute for Emery.* B. Hess, Bayreuth. Eng. Pat. 2321, 1883.

THE materials to be used under this invention are "serpentine (or kindred minerals), feldspar, mica, quartz, and fireclay, or some of these variously combined, according to the purpose for which the artificial stone is intended." They are finely ground, mixed in the desired proportions, "moistened with so much water that the mass will remain conglomerated if compressed by hand," and the moist mixture is then compressed in moulds, so that when removed from the moulds it will retain the form given to it by them. "The so-formed stones, plates, vessels, or other objects, are afterwards well but slowly dried under the influence of a gradually increasing temperature, and are finally burned in a strong white heat."

## X.—METALLURGY, MINING, Etc.

*On the Modern Methods of Manufacturing Iron and Steel, on the Methods of testing these Materials, and their Characteristic Properties.* Franz Kupelwieser. Dingl. Polyt. Journ. 250, 37, reprinted from the "Wochenschrift des Oesterreichischen Ingenieur- und Architektenvereins," 1883, 187-191.

THIS paper deals with the difficulties which manufacturers of iron and steel, especially steel rail makers, have to contend with to satisfy the conditions specified by consumers with regard to the quality, dimensions, etc., of the articles manufactured. The various methods for making malleable iron and steel from pig iron are first briefly described, dividing the processes roughly into two classes, those in which the temperature is not so high that the metal is in the fluid state at the end of the operation, as in the ordinary puddling furnace and the charcoal hearth finery, and those in which the purified iron is in a fluid state at the end of the process, as in the crucible steel process, the Bessemer process and the Siemens-Martin process. One of the chief advantages of the latter class is that the slag and cinder are more completely separated from the metal. It is also a fact that melted steel absorbs large quantities of gases, which have been found to be principally hydrogen and nitrogen. These occluded gases have the tendency to form bubbles in the ingots, and must of course tend to prevent the metal being, in subsequent treatment, rolled or welded to absolute uniformity of physical structure. The Siemens-Martin process in this respect has an advantage over the Bessemer process, in that less gas is likely to be occluded by steel made by the Siemens-Martin method than by the Bessemer method. As regards the chemical influence of these occluded gases, the question has not yet been satisfactorily decided. The author then discusses the causes affecting the characteristic qualities of the manufactured steel, and classifies them as follows: (1) Chemical composition, (2) homogeneity of the ingot steel, and (3) the mechanical treatment. He then describes briefly the effect that the various elements found together with iron in steel have upon the physical properties of steel, and quotes the experiments of Stubbs and Snelus, showing that these elements are unequally distributed in an ingot of cast steel. He then speaks at some length of

the influence of mechanical treatment. By this the quality can be either improved or deteriorated. By working the material down from a piece of large cross section to a piece of small cross section, the tendency of the operation is to improve the quality. It is a known fact that by a sufficiently prolonged mechanical treatment steel can be made to bear a greater tensile strain. The process of reducing the area of the cross section of a piece of steel tends to make the material denser, to squeeze the sides of bubbles in the ingot closer together, and, when a proper temperature is maintained, to weld the walls of such bubbles together, to make the texture of the metal finer grained, and, assuming that the mechanical treatment is continued during a gradual fall of temperature in the metal, to make the breaking strain greater. If, on the other hand, the section at the beginning of the operation is not one suitable to the machine employed, the choice of a too large section may become as great an error as the choice of too small a section, as, in order to obtain the final section required, the metal has to be worked towards the end of the operation at too low a temperature. The reason why rails from Austrian works are weaker than those made in German, in spite of the unrolled steel being at least as good if not better in Austrian works than it is in German, is that in Germany there is a more perfect arrangement in the works, enabling a greater reduction of sectional area by mechanical treatment to be effected. The unequal distribution of the metal in the section of the finished product has also a great influence upon the resulting quality of the manufactured article. Those portions where there is most metal remain the hottest, whilst the thinner portions get more rapidly cool, and are thus worked at a lower temperature, and become therefore harder and also more brittle. The harder the material is to begin with the worse this feature becomes. In rail-making, for instance, the head of the rail contains more metal than the foot, and hence the foot at the end of the rolling is colder than the head, and is therefore harder, whereas it would be more desirable to have the softer metal in the foot rather than in the head. The effect of unequal cooling can be seen by the eye in almost every broken rail-section. Sometimes a rail breaks without any apparent reason. Often the cause is to be found, not in any imperfection of material or manufacture, but in the fact that the section of the rail was designed without any consideration being given to the peculiar properties of the metal of which it was to be made, and simply because that section gave the greatest calculated carrying power for the smallest quantity of material. The author lays stress upon the point that it is not possible in the manufacture of an article on a large scale to obtain a product perfect from all theoretical points of view, since it is impossible to heat the material to exactly the same temperature in all its parts, and in removing it from the furnace, and during its mechanical treatment, heat is unequally lost, so that the mechanical treatment cannot be conducted under equal conditions of temperature, and hence the same article shows different properties when tests are applied to different parts of it. Beyond this, it must be borne in mind that the machines used are not instruments of precision, since they require the expenditure sometimes of thousands of horse-powers, and since they have to deal with iron at a high temperature. All idea of working with precision ends when one has to deal with hot metal, since with every change of temperature in the metal being rolled, changes in the size of the rolls take place, and with these, changes in the measurements of the manufactured article. Of course, more perfect machinery makes a more perfect article; but it is sometimes striking that the requirements of consumers go so far as to be beyond attainment, even with the best materials and the most careful work, and that among the requirements are some which depend upon or demand properties totally inconsistent with those necessary to satisfy others. The author then instances some cases in which measurements are demanded for rails which cannot possibly be uniformly attained, and with regard to which little toleration is allowed; other cases in which a certain chemical constitution is required, the rails being rejected unless this be satisfied, although they may for all practical purposes be excellent



in quality and satisfy all breaking or tensile tests. He concludes by remarking that by the requirements of the consumers, by the difficult conditions of contract, by the number of different sections demanded by different consumers, necessitating a corresponding number of sets of rolls, and by the inconveniences of the contract itself, the cost of manufacture is increased, and therefore also the selling price, which can hardly be a matter of small importance to the purchaser.—H. S. P.

*Improvements in the Method of and Apparatus for Depolarising Electric Baths in the Refining and Deposition of Metals.* A. M. Clark, London. Communicated by C. de Changy, Levallois-Perret, France. Eng. Pat. 2281, 1883.

THIS invention consists in a method of "automatically depolarising electrolytic baths, which method consists in breaking, diminishing, or increasing the contact of the main current, and making, approaching, or receding from a contact with the circuit of the electrolytic bath or baths with or without breaking the main circuit."

*Improvements in the Manufacture of Nickel and of Cobalt, and of Alloys of either of these Metals with Iron.* A. M. Clark, London. Communicated by the Société Anonyme Fonderie de Nickel et Métaux Blancs, Paris. Eng. Pat. 2243, 1883.

THIS invention consists in melting together, "in the same crucible, metallic nickel or cobalt with cyanide or ferrocyanide of potassium (with the addition of iron when desiring to produce ferro-nickel and ferro-cobalt) in presence of an oxide of manganese." "By treating nickel, cobalt, ferro-nickel, and ferro-cobalt with oxide of manganese and cyanide or ferro-cyanide of potassium, with or without the addition of a very small proportion of aluminium, I not only obtain metals and alloys suitable for rolling and drawing, but I am enabled to obtain castings of these metals and alloys (which has never hitherto been practically possible) suitable for the manufacture of surgical instruments, cocks and valves, locksmiths', carriage smiths', and gunsmiths' work, etc."

*A New or Improved Process for Manufacturing Metallic Oxides or Bases.* H. A. Bonneville, Paris. Communicated by L. C. E. Fancheux, Loos, France. Eng. Pat. 2055, 1883.

THIS patent claims the "principle" of decomposing the metallic sulphides by steam, superheated or not.

*Improved Processes for Obtaining Aluminium from its Ore or from other Substances.* H. A. Gadsden, London. Communicated by E. Foote, New York. Eng. Pat. 1995, 1883.

THE author of this invention proposes to treat either double chloride of aluminium and sodium or double fluoride of aluminium and sodium by vapour of sodium, evolved from retorts in which a mixture of carbonate of soda with carbonaceous matter is intensely heated.

*Improvements in the Use of Slags or Scoriar Obtained as Waste Products from the Manufacture of Steel by the Converter Process, etc.* J. Wright, London. Eng. Pat. 1657, 1883.

THE object of this invention is to recover from converter slags the "small masses of steel and iron, in different metallic states and in volumes varying from a very small shot up to the size of a walnut," which the inventor states to sometimes constitute 20 per cent. of the weight of such slags. He crushes the slags, in such wise that the other constituents thereof are reduced to powder while the said small masses of metal "resist disintegration and retain their volume," and he then separates the latter "by means of sieves of various meshes."

*Improvements in the Method of and Apparatus for Manufacturing Metallic Alloys, more particularly those of Zinc.* G. Selve, Altena, Germany. Eng. Pat. 2995, 1883.

IN order to diminish loss of zinc by volatilisation in the manufacture of brass, and to prevent the workmen employed in that manufacture having to be exposed "to a high degree of heat and the injurious fumes of zinc," the author of this invention proposes to employ in that manufacture specially deep crucibles, fitting into apertures in the arch or roof of a regenerative furnace in suchwise that only the lower part of each crucible descends into the furnace and is heated thereby, the upper part of the crucible extending above the roof of the furnace, and so being exposed to the atmosphere. "Copper is first placed in the crucible, and is melted at a temperature of from 1,922° to 2,192° Fahr., and the zinc is then quickly added, . . . so much brass waste being then added as to render the whole pasty, while at the same time cooler layers of metal are by this means formed in the upper part of the crucible. As, in addition to this, the upper part of the crucible is outside the furnace, the upper layers of metal are of a much lower temperature than those below, and they are maintained at this low temperature by successive further additions of cold brass waste, thereby causing the condensation of the greater part of the zinc vapours as they rise up into the cooled layers from the lower highly-heated layers of metal. After the whole of the metal is melted, the crucible is removed and the contents stirred, so as to obtain a homogeneous alloy." The inventor states that the loss of zinc incurred in the manufacture of brass by this method is from 50 to 75 per cent. less than the loss of zinc in the ordinary method of manufacturing that alloy.

*Improvements in the Utilisation of Phosphatic Metallic Scoriar.* G. Pitt, Sutton. Communicated by G. Récour, Liège, Belgium. Eng. Pat. 1743, 1883.

BY this invention the slags produced in the basic process of manufacturing steel are to be fused in a cupola or blast furnace in a reducing atmosphere. The slags "should contain or be mixed with such fluxes as to contain as a whole about 3 to 4 parts of iron and manganese together for 1 part of phosphorus, and 30 to 50 of silica for 50 to 70 parts of alumina, lime, and magnesia together. The fuel should be coke, anthracite, or charcoal, and the temperature high enough to reduce a large portion of the manganese oxide" contained in the slags operated upon. The products are said to be "a phosphoric matte, with 20 to 25 per cent. of phosphorus, and a slag very poor in phosphoric acid." Two methods of dealing with the phosphoric matte are described. (1) The matte "is blown in a basic converter as ordinary pig iron, with the addition of 2 to 4 parts of lime or calcined dolomite for 1 part of phosphorus in the matte. . . . On leaving the converter before complete dephosphorisation of the metal, a slag very rich in phosphoric acid and rather poor in manganese and iron can be run, which slag can be treated by sulphuric acid for superphosphate for agricultural purposes." The metal remaining in the converter can then be completely dephosphorised in the usual way. (2) The phosphoric matte "is ground and mixed with 3 to 7 parts of sulphate of soda or potash for 1 part of phosphorus in the matte, and about one half part of coal or coke." The mixture so obtained is then heated in a reverberatory furnace, first in a deoxidising atmosphere, and then, after a certain time, "under an oxidising flame, and the temperature raised as high as possible." The products are phosphate of soda or potash and sulphides of iron and manganese. The former is then separated from the latter by lixiviation.

*Treating Blast-Furnace Slag to obtain therefrom certain Products useful for various purposes.* E. G. Colton, London. Communicated by A. D. Elbers, Hoboken, U.S.A. Eng. Pat. 2252, 1883.

BLAST-furnace slag is first to be converted into "slag-fibres, known as slag-wool, mineral wool, and silicate cotton," and the said slag-fibres are then to be "heated

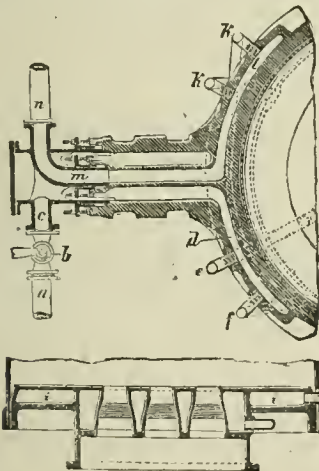
and fritted" until "they are reduced to white friable silicates, which are then pulverised." The product so obtained is said to be suitable for use (1) "As a flux or glaze for china, pottery, and enamelled ware, or as the body or main constituent in making new kinds of pottery and similar ware. (2) The manufacture of glass. (3) In making ornamental plastering, stucco, statuary, and artificial stone, mixed with lime, in the same manner as these products are now made from fine cements. And (4) as a paint or body for pigments."

*Preparation of Basic Bricks from Strontianite.* G. Stöckman, Ruhrort. Ger. Pat. 24226, Sept. 21, 1882.

THE raw or burnt strontianite is mixed with clay or argillaceous ironstone in such proportions that the bricks shall contain about 10 per cent. of silica, and then it is worked into a plastic mass with tar or heavy hydrocarbons, and pressed into bricks. After coming out of the moulds the bricks are dusted with fine clay or ironstone, dried, and burned. The effect of dusting is that during the burning the clay combines with the strontia and forms a glaze which protects the bricks from the moisture in the air.—W. M.

*Water Cooling for Bessemer Hearths.* Willy Schmachtenberg, Hoerde. Ger. Pat. 23309, Aug. 16, 1882.

THE water passes through an annular space in the converter-trunnion, as shown, to a canal *d* in the con-



verter-ring; from thence, by tubes *c f*, to water spaces *i* in the tuyere plate. From here the water returns by the tubes *k k* to a central pipe in the trunnion, and thence, as shown by elbow-pipe *m*, to the waste pipe *n*.—J. T.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*Refining, Deodorising, and Purifying Oils and Fats, etc.* A. C. Tichenor, Alameda, U.S.A. Eng. Pat. 2229, 1883.

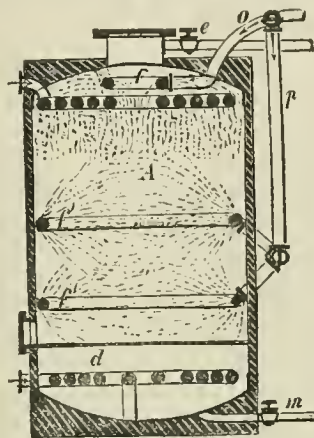
THE various objects of this invention are to be accomplished by passing electric currents through the bodies to be refined, deodorised, and purified.

*An Improved Process for the Manufacture of Vegetable Oils.* C. F. Stollmeyer, London. Eng. Pat. 2334, 1883.

THIS invention consists in "breaking or bursting the oil cells" of seeds by heating them in closed vessels with water under a pressure of 75 lb. to the square inch. The resulting mixture of water, oil, and pulp which is then discharged from the said vessel is allowed to stand till all the oil has collected at the top. The supernatant oil is then drawn off, and the pulp is then submitted to pressure for the extraction of the oil still remaining in it. The inventor claims that this process gives "a greater yield of oil and a purer and better article" than can be obtained by the processes at present in use.

*Apparatus for Extracting Fat under pressure from Bones and other Materials.* Werner Schneider, Lehrberg, near Aushach. Ger. Pat. 22295, May, 1882.

THE bones having been filled in, some petroleum spirit is introduced through the pipe *m* and raised to boiling by means of steam which passes through the worm *d*. The vapours pass through *e* into the condenser. When



the bones are sufficiently heated and the air is expelled from them, cold petroleum spirit is injected under strong pressure through the pipes *o* and *p*, and the roses *f*<sup>1</sup>. The spirit in *A* is then heated again. Part of it is thus evaporated, and is then forced back under pressure through the roses. These operations are repeated several times. The claims of the patentee refer to the use of the perforated pipes *f*<sup>1</sup>, which are placed at the sides of the apparatus, and below the surface of the layer of bones, in order to force the solvent also from the side into the materials which are to be extracted.—J. G.

*A Process of Decomposing and Transforming Fats or Fatty Substances into Fatty Acids and Glycerine.* A. Marix, Paris. Eng. Pat. 2349, 1883.

By this invention fatty substances are digested with water and "a neutral substance acting purely as a mechanical agent for opening up the molecules and breaking through the albuminous film surrounding the same." The said neutral substance may be "carbonate of magnesia, tale, pipeclay, chalk," etc. The proposed mode of procedure, and special apparatus for it, are described in detail.

*New or Improved Process for Purifying Glycerine.* H. J. Haddan, London. Communicated by C. Moldenhauer and Dr. C. Heinzerling, Frankfurt-on-Main. Eng. Pat. 2326, 1883.

GLYCERINE from soap-makers' leys is usually contaminated by common salt, and thereby rendered unfit for some of the purposes to which glycerine is applied in the arts. The main object of this invention is to purify glycerine from salt. The crude glycerine is first "completely separated from water," and is then "mixed with once or twice its volume of pure methyl or ethyl alcohol, and, if this mixture be not alkaline, from 1 to 10 per cent. of carbonate of soda is added." Certain salts precipitate, and are then separated by filtration. Sulphuric acid is then added to the mother-liquor until it has an acid reaction. The sulphuric acid decomposes the various sodium salts of organic acids present in the mother-liquor, and the resulting sodium sulphate separates out, and is removed by a second filtration. The filtrate "is now mixed with lead oxide or soluble lead salts or other metallic salts which form insoluble compounds with chlorine, in sufficient quantities to precipitate the latter." The resulting precipitate having been removed by a further filtration, the final filtrate is distilled, to drive off its alcohol. "At the end of this operation, vapours of ether pass over, which have been produced from the volatile



organic acids, such as propionic and butyric acids, contained in the ley, in the presence of alcohol and acid. The treatment with alcohol, therefore, serves to remove, not only the common salt, but also, in the shape of volatile mixed ethers, the volatile fatty acids contained in the crude glycerine, which acids are very prejudicial, especially in the manufacture of nitro-glycerine."

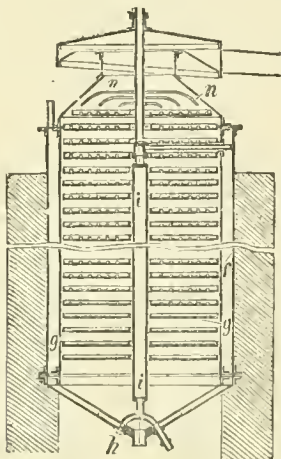
*An Improvement in the Treatment of Fats for the Manufacture of Soap.* J. Imray, London. Communicated by J. A. F. Bang and J. de Castro, Paris. Eng. Pat. 3658, 1883.

"FATS are frequently subjected to the action of heated water with a little zinc powder in a close vessel, for separation of the glycerine before saponification. The fatty acids act on the metal of the vessel, producing compounds which, mingled with the fat, seriously discolour the soap produced from it. This invention relates to means of preventing this discolouration. For this purpose, the fatty matter, after being treated in the usual way for separation of the glycerine, is poured into a vessel lined with lead, and while still hot it is treated with sulphurous acid applied either in solution with water, or blown through the material to which water is added. The sulphurous acid acts on the metallic compounds, forming new compounds which are soluble in water, and can be drained off with the water, and it also to a certain extent bleaches the fatty matter, especially that which is of vegetable origin, so that the soap produced from it is free from objectionable discolouration."

## XII.—PAINTS, VARNISHES, AND RESINS.

*Improvements in the Process and Apparatus for the Preparation of Clear Amber Varnish.* Bernhard Borowsky, Stargard, Pomerania. Ger. Pat. 21909, July, 1882.

THE molten amber, which has been mixed with oil, is diluted with a considerable quantity of turpentine, and put away for several weeks, when all impurities will settle out. The clear solution is siphoned off, and placed



in a distilling apparatus, in which the excess of the solvent is distilled off by means of air, which is blown through. The remaining mass has the consistency of syrup, and is diluted with turpentine before being used. The apparatus contains perforated rimmed plates *g*, the air passing through slits which are in the latter. The apparatus is heated by means of the steam casing *f* and the pipe *i*. The oil enters through pipes *n* and runs off at *h*. The air enters at *h*, and leaves at the top, carrying away the vapours.—J. G.

## XIII.—TANNING, LEATHER, GLUE, AND SIZE.

*An Improved Process for Removing the Hair or Wool from the Skins or Hides of Animals, and also for Cleansing, Softening, Swelling, and Preparing the same for subsequent Treatment in the Production of Leather.*

THIS invention "consists in subjecting hides or skins alternately to the action of water and to that of the open air."

## XIV.—AGRICULTURE, MANURES, Etc.

*Process for Destroying the Vine Louse and other Injurious Insects by the Application of Pyridine Bases.* Julius Rütgers, Vienna. Ger. Pat. 24295, Nov. 30, 1882.

THE pyridine bases are obtained from the portion of coal-tar oil boiling between 80° and 250° C. by agitating it with a mineral acid and then treating the acid extract with soda ley and distilling with steam. The bases or their aqueous solution are syringed either on to the vines or on the soil, or are blown in the form of vapour in the freshly turned up earth. When salts of the pyridine bases are used, decomposition generally takes place by the action of the alkaline earth carbonates in the soil, the bases being liberated, or this decomposition is effected by spreading lime or carbonates on the ground. This mixture of bases acts very energetically and lasts long, owing to the vapour being retained in the ground.—W. M.

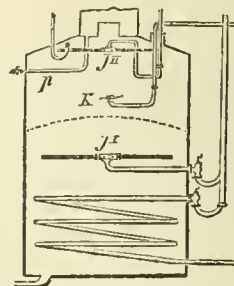
*Improvements in the Manufacture of Fertilisers.* H. J. Allison, London. Communicated by the Scribner Process Company, New York. Eng. Pat. 4036, 1883.

THE authors of this invention calcine ferruginous and aluminous phosphates, "bringing into contact with the phosphates, while hot, either the vapour of sulphur or sulphurous anhydride." They state that the product is "a commercially valuable fertilising compound, containing a large percentage of available phosphoric acid," and that it may "without further treatment be mixed with potash salts, or ammoniacal matters," valuable manures being thus produced without the use of sulphuric acids.

## XVI.—BREWING, WINES, SPIRITS, Etc.

*Method and Apparatus for Converting Ordinary Alcohol Distilling and Rectifying Apparatus into continuous ones.* J. A. Stelzner, Alchemnitz, near Chemnitz, Saxony. Ger. Pat. 22464, March 26, 1882.

THIS method has for its object the production of pure spirit by one distillation. The crude spirit, which has previously been mixed with certain materials serving for



its purification (oil, acids, alkalis, alkaline carbonates, etc.), is introduced into the still by means of the spray apparatus *K*, which is kept in continual action by an injector. Here it mixes with steam, escaping from the

spreading pipes  $J_1$  and  $J_2$ , whereupon the mixed vapours ascend into the rectifying column. In this manner a uniform low temperature is maintained, and therefore no heavy vapours are carried over. The latter accumulate in the lowest partition of the rectifying column, from which they are carried off through the pipe  $p$  into another small column, in which they are further separated.—F. M.

*An Improved Process of Purifying Water and adapting it for Brewing and other purposes, and Apparatus therefor.* E. M. Dixon, Glasgow. Eng. Pat. 2112, 1883.

Two methods of purifying water are described in the specification of this invention. One "consists in adding slaked lime to the water and injecting air, then separating the sediment, and subsequently injecting carbonic acid into the water;" the other in "first dissolving in the water slaked lime and injecting air while a high temperature is maintained, then adding hydrochloric acid or sulphuric acid or other suitable reagent, and subsequently injecting carbonic acid, and filtering or decanting."

## XIX.—PAPER, PASTEBOARD, Etc.

*An Improved Process for Sizing Paper and Textile Fabrics, which is also applicable to the manufacture of Parchment Paper, Leather Boards, etc.* C. Weygaug, Childs Hill. Eng. Pat. 2251, 1883.

THE sizes to be used under this invention are starch and animal glue. With them are to be used various "astringent metallic salts," as sulphate of zinc, sulphate of alumina, and sulphate of magnesia. Various modes of procedure are described. In some of them there is combined with the "sizing" of paper pulp the precipitation among it of "filling" material, such as sulphate of barium.

## XXI.—EXPLOSIVES, MATCHES, Etc.

*Improvements in the Manufacture of Explosive Compounds.* F. W. Gilles, Köln, Germany. Eng. Pat. 1883, 1883.

THE author of this invention proposes to manufacture explosive compounds by treating molasses by a mixture of nitric and sulphuric acids. By one method he mixes 380 grammes of raw molasses, 1,000 grammes of fuming nitric acid, and 2,000 grammes of "concentrated sulphuric acid," and washes the product several times, alternately with cold and then with warm water. "The compact nitro-molasses thus obtained is of either a gray, yellow, or whitish colour, and can be used at once as an explosive material, or it can be added to any absorbent and formed into blasting cartridges. A more powerful explosive in the fluid form is obtained from the molasses, if the same," after having had as much water as possible evaporated off from it, "is brought, by being treated with brown peroxide of lead and sulphide of carbon, or by passing oxygen through the mass, or by addition of oxygen, to a composition corresponding to 30 per cent. carbon, 20 per cent. oxygen, and 50 per cent. water chemically combined, the mineral components not being" counted. It is when the molasses contains more than the just stated proportion of oxygen that it is to be treated by peroxide of lead and bisulphide of carbon; when it contains less than that proportion of oxygen it is to be treated by oxygen gas, forced into it under pressure in closed vessels. "In all cases, the proportion of carbon to oxygen should be in the ratio of 3 to 2." When the molasses is treated by peroxide of lead and bisulphide of carbon, these bodies are added to the molasses in open vessels, and the mixture is allowed to ferment, for periods varying, with the temperature, from 24 hours, which are sufficient when the mixture is maintained at a temperature of  $70^{\circ}\text{C}$ ., to 28 days, which is

the period required when the temperature is below  $18^{\circ}\text{C}$ . The "prepared" molasses is then nitrated as above described for raw molasses. "The fluid nitro-molasses thus obtained when heated slowly boils at between  $180^{\circ}$  and  $200^{\circ}\text{C}$ ., and detonates between  $220^{\circ}$  and  $250^{\circ}\text{C}$ . It can be mixed with any absorbents, as for instance," kieselgühr. For blasting purposes, the inventor recommends various mixtures of nitro-molasses with sawdust, tan, or bran which has been previously impregnated with nitrate of potash.

*Improvements in the Manufacture of Explosive Compounds.* E. Turpin, Paris. Eng. Pat. 2139, 1883.

THE object of this invention is to manufacture "an explosive compound which acts similarly to ordinary gunpowder, but with about three times greater power, provided the ignition of the charge be effected simply by fire, or which produces powerful explosions even in the open air, similar to those produced by dynamite, if the ignition of the charge be effected by the initial explosion of a suitable detonator, such as one having a priming consisting of a grain of fulminate of mercury." The said explosive compound is formed by mixing together about 80 parts of pulverised chlorate of potash and 20 parts of ordinary coal tar, adding from 1 to 10 per cent., according to the degree of fluidity of the tar used, "of a suitable porous absorbent substance, such as powdered wood charcoal, silica, kieselgühr, randanite, and the like." "If it is desired to increase the sensitiveness of the compound, from 1 to 10 per cent. of permanganate of potash is substituted for from 1 to 10 per cent. of the chlorate of potash;" in the reverse case, for part of the chlorate of potash nitrate of potash is substituted.

## XXII.—GENERAL ANALYTICAL CHEMISTRY.

*On the Estimation of Carbon in Cast Iron and Steel.* G. Zabudsky. Journ. d. russ. phys.-chem. Gesellschaft 1883 (1) 410; and Berl. Ber. 16, 2318.

IN place of a solution, the author employs for the decomposition of carboniferous iron a dry mixture of copper sulphate and sodium chloride, which is prepared by the evaporation of 100c.c. of a solution containing 20 grms. of each salt. For every gram of finely-divided iron 20 grms. of the mixture are taken; enough water is added to form a pasty mass when stirred, and the whole is triturated in a glazed mortar until hard particles are no longer detected under the pestle. The pasty mass is then brought into a glass vessel, the mortar rinsed with a solution of ferric chloride, and the mixture gently warmed, at first alone, and finally with hydrochloric acid. The residue of pure carbon is then transferred to an asbestos filter. The whole operation takes from 2 to  $2\frac{1}{2}$  hours. The combustion of the residue gives perfectly satisfactory results. S. Y.

## New Books.

**BLEACHING, DYEING, AND CALICO PRINTING.** With Formule. London: J. and A. Churchill, 11, New Burlington Street. 1884. (One of Churchill's Technological Handbooks.) Edited by Mr. JOHN GARDNER.

THE book is an octavo volume, bound in cloth, and contains 203 pages and five illustrations. The matter is distributed in four chapters, these being followed by a short appendix. Chap. i., bleaching, 33 pages; Chap. ii., dyeing, 28 pages; Chap. iii., calico printing, 22 pages; chap. iv., dyestuffs, 58 pages; the appendix occupying two pages, and the alphabetical index seven pages. Since in this small work the attempt is made to deal with dyestuffs, as well as their employment in the tinctorial arts, it is scarcely necessary to say the treatment of the



subject is very concise. Receipts are proportionately somewhat numerous, and, in the main, the object of the work seems to be to give concise practical rules for bleaching, dyeing, and calico printing, though here and there opinions on certain specific points are given and supported by authoritative evidence, and these opinions will be doubtless of considerable value.

TECHNISCH-CHEMISCHES JAHRBUCH, 1882-1883: Ein Bericht über die Fortschritte auf dem Gebiete der chemischen Technologie von Mitte 1882 bis Mitte 1883. Herausgegeben von Dr. RUDOLF WIEDERMANN. Fünfter Jahrgang. Mit 337 in den Text gedruckten Illustrationen. Berlin: Verlag von Julius Springer, 1884.

AN 8vo volume, bound in cloth, 608 pages and 337 wood engravings, tables of contents at the beginning, and double alphabetical index of names of authors and of subject matter at the end. The price of the work is 12m. (12 shillings). As regards its plan, it is a model of skilful arrangement, and follows, to a considerable extent, the method of the German Patentblatt und Die Chemische Industrie. The contents are arranged in the following order: (A) General literature of statistics. (B) Chemical technology. Metallurgy, pages, 10 to 62; salt and soda, 63 to 73; potassium compounds, 89 to 90; ammonia, 91 to 98; alkaline earths, 101 to 107; alum, 110 to 114; glass, 114 to 125; pottery, 125 to 151; building materials, limes, cements, etc., 151 to 166; explosives, 166 to 184; fuel and gas, 184 to 228; sugar, 228 to 273; starch and starch-sugar, 273 to 286; wine, 286 to 303; beer, 303 to 325; spirits, 325 to 357; fats, oils, soap, etc., 357 to 373; water, 373 to 400; foods, etc., 400 to 436; manures, 436 to 445; tanning, 445 to 456; glue, 456 to 458; dyestuffs, inorganic colours, and pigments, 458 to 462; tar-colours, 462 to 500; textiles, 500 to 504; paper, 504 to 511; photography, etc., 511 to 515; organic acids, 515 to 518; alkaloids, 518 to 525; apparatus, 525 to 546. Review of new books in the period stated, 579 to 597. This Bücherschau (review of new books) is certainly a novel and useful feature in a work of this kind.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1883.

5832 N. L. Pouschkareff, Moscow. Improvements in the means and apparatus for lighting by means of hydrocarbon vapour, part of which apparatus is also applicable for storing inflammable liquids generally. December 21  
5841 C. D. Abel, London. Improvements in desiccating woods. Communicated by J. A. Koch, Galveston, U.S.A., and W. Herre, Berlin. December 22  
5846 T. W. B. Mumford and R. Moody, Victoria Dock. Improvements in apparatus for grinding, crushing, or reducing to powder ores, quartz, or other like substances. December 22  
5847 A. P. Price, London. Improvements in recovering and obtaining tin from tinned metallic surfaces. December 22  
5849 P. M. Justice, London. Improvements in the manufacture of alkalis and alkaline salts. Communicated by S. G. Thomas, Algiers. December 22  
5855 C. D. Abel, London. Improvements in the impregnation of wood and textile fabrics with protective or preservative substances. Communicated by J. A. Koch, Galveston, U.S.A., and W. Herre, Berlin. December 24  
5851 G. Hand-Smith, Chelsea. Improvements in sinking or fixing colours, marks, or designs into or on marble, wood, ivory, and other materials, and in apparatus employed therein. December 26  
5862 G. Hand-Smith, Chelsea. Improvements in the manufacture of white lead and in apparatus employed therein. December 26  
5867 H. Brunner, Widnes. Improvements in the manufacture of baskets, skeleton barrels, or crates for carrying acid carboys or other merchandise requiring a strong but not water-tight casing or package. Communicated by C. Garneri, Paris. December 27  
5868 W. Weldon, Burstow. Improvements in and relating to the manufacture of chlorine. December 27  
5873 N. McFarlane Henderson, Broxburn. Improvements in obtaining oil and gas from minerals, and in apparatus therefor. December 27

5878 J. Jameson, Newcastle-on-Tyne. Improvements in the preparation of safety paper. December 27  
5886 F. C. Glaser, Berlin. Improvements in the process for treating zinc ores. Communicated by G. v. Kramsta'sche Erben and R. Wiester, Kattowitz, Germany. December 28  
5891 J. Fordred, Tottenham. Improvements in the manufacture of German or dried yeast, part of which improvements is applicable to the production of vinegar. December 28  
5893 J. Inray, London. An improvement in apparatus for evaporating, distilling, or concentrating liquids. Communicated by H. Egells, Berlin. December 28  
5903 J. McCulloch, and H. McVicar, Calderbank, N.B. Improvements in obtaining oil and other products from minerals and in apparatus therefor. December 28  
5914 G. A. Diek, London. Improvements in the manufacture of metallic alloys or compounds. December 29  
5938 E. Morewood, Llanelly. Improvements in coating metal plates. December 31  
5954 W. Green, St. Lawrence. Improvements in the treatment of mineral oils in combination with other matters, and producing therefrom soap, candles, and other articles. Dec. 31  
5958 C. F. Claus, London. Improvements in obtaining from alkali-waste either free sulphur, or free sulphur and sulphurous acid, or free sulphur and sulphuretted hydrogen. December 31  
5959 C. F. Claus, London. Improvements in obtaining sulphur from sulphuretted hydrogen. December 31  
5960 C. F. Claus, London. Improvements in the treatment of mixtures of sulphuretted hydrogen and other gases to obtain the sulphur of their sulphuretted hydrogen as sulphurous acid. December 31  
5962 H. J. Haddon, London. An improved process and apparatus for the manufacture of white lead. Communicated by F. Schmoll, Cologne. December 31  
5965 G. W. von Nawroeki, Berlin. Improvements in bleaching leather and tanned hides. Communicated by G. Levinstein, Wiesbaden. December 31  
5969 R. Baynes and J. Fearnside, jun., and W. P. Thompson, Liverpool. Improvements in the bleaching or purification of petroleum, and of other distillable oils. December 31  
5974 C. B. Warner, London. Improvements in the manufacture of artificial bone, ebony, marble, porcelain, and other substances. Partly communicated by Ljnbomir Mestaniz, New York. December 31  
5979 A. J. Boulton, London. Improvements in regenerative coke furnaces. Communicated by the Schlesische Kohlen und Koks Werke, Gottesberg, Germany. December 31

1884.

16 J. Haason, Biogley. Improvements in apparatus for extracting ammonia and other impurities from coal gas. January 1  
32 H. H. Lake, London. An improved compound for plastering or stucco work. Communicated by H. E. Scales, Newton, U.S.A. January 1  
48 J. G. Willans, West Hampstead. Improvements in the manufacture of aluminium. January 1  
49 J. G. Willans, West Hampstead. Improvements in obtaining volatile products from coal. January 1  
71 J. Armstrong, Clapham. Improvements in the manufacture of beer or other beverages, and extracts or products to be employed in such manufacture, and for similar purposes. January 1  
72 A. C. Henderson, London. An improved process for bleaching rags, tissues, and textiles of all kinds, and paper pulp. Communicated by L. de Ronssen, Paris. January 1  
92 S. C. Dean, Fulham. Purifying sewer gas and mephitic vapour. January 1  
93 S. C. Dean, Fulham. Purifying sewage and foul waters. January 1  
94 R. Nicholls, Hendon. Improvements in the treatment with sanitary objects of human excreta and other putrescible matter, and in apparatus and means employed in the conversion of the same for manurial purposes. January 1  
143 B. C. Molloy, M.P., London. Improvements in amalgamating gold and other metals, and in the apparatus employed therein. January 1  
151 J. H. Johnson, London. Improvements in the manufacture of paper. Communicated by J. A. Mayraels, Paris. January 1  
153 R. Stone, London. Improvements in the manufacture of cement, bricks, tiles, and similar articles, which are also applicable for smelting, heating, and burning purposes generally, and in machinery and apparatus for carrying the same into effect. January 1  
203 H. R. Snelgrove, Clapham. Improvements in apparatus for purifying and increasing the illuminating power of coal gas. January 1  
209 H. J. Haddon, Westminister. A new or improved process for producing assimilable phosphates from phosphoric slags or residues obtained in the manufacture of iron and steel. Communicated by A. W. B. S. van Oldruitenborgh, Liège, Belgium. January 1  
221 D. Urquhart, Westminister. Improved means for suspending the corrosive action of acids and acid mixtures, more particularly applicable to the manufacture of disinfecting compounds and polishing powders. January 1  
222 Professor A. K. Huntington, London. An improvement in the manufacture of tungstic acid and its compounds. January 1  
221 Professor A. K. Huntington and J. Humphrys, London. An improvement in the treatment of scrap zinc for the recovery of zinc and solder. January 1  
338 C. M. Pielticker, Kilmarnock. Improvements in the production of steel and other metal wire, plates, rods, bars, and

rails, and also in apparatus connected therewith. Partly communicated by Dr. F. C. G. Müller, Brandenburg, Germany. January 2

351 H. Knowles, Woodville. Improvements in continuous and semi-continuous action kilns for burning bricks, pottery-ware, or lime, or for other like purposes. January 2

352 H. Knowles, Woodville. Improvements in ovens or detached kilns for burning bricks, potteryware, or lime, or for other like purposes. January 2

418 C. J. Mountford, Birmingham. Improved manufacture of fire-proof paints. January 2

443 C. E. Bell, Durham. Improvements in apparatus for coking and distilling coal. January 2

474 H. Knowles, Woodville. Improvements in fireplaces for heating kilns, pottery ovens, glass furnaces, or other heating purposes. January 2

475 H. Knowles, Woodville. Improvements in the construction of kilns, pottery or coke ovens, glass furnaces, or like structures. January 2

491 B. P. Walker, Birmingham, and J. A. B. Bennett, King's Heath. Improvements in self-acting retorts for the distillation of solid substances. January 3

493 J. Illingworth, Batley. An improved method of and apparatus for treating fibres, rags, and woven fabrics. January 3

551 L. A. Groth, K.G.V., London. A new and improved process and apparatus for the extraction of metals from their alkaline combinations. Communicated by R. Grätzel, Hanover, Germany. January 3

552 L. A. Groth, K.G.V., London. A new and improved method of extracting biphosphates from the phosphated slags that are produced by the basic Thomas and Gilchrist process of making fluid steel. Communicated by R. Schliwa and A. Schliwa, Dortmund, Germany. January 3

579 M. Williams, Wigan. Improvements in the manufacture and preparations of drying oils. January 3

603 Sir F. C. Knowles, Bart, Ryde. A new or improved method of treating sulphides and oxides of metals and obtaining products therefrom. January 3

637 W. E. Leyland, St. Helens. Improvements in the construction of apparatus for consuming smoke and sulphurous or other noxious vapours. January 4

709 E. Crewe, Middlesbrough. Improvements in apparatus for controlling and regulating the production of gas in gas producers. January 4

727 J. Kay, Bury. Improvements in the process and apparatus for manufacturing white lead. January 5

751 J. Wheeler, Hiracombe. Improvements in the manufacture of miscible tar. January 5

753 J. Sansom, Glasgow. An improved oil compound for use in quelling waves at sea. January 5

761 R. Cooper, Over Darwen. Improvements in apparatus for straining pulp used in the manufacture of paper. January 5

761 A. G. Southby, Jersey. Improvements in the mode of and apparatus for cleansing beer. January 5

774 G. Dunbar, Malam, Bingley. Improvements in washers for the purification of gas, which apparatus is to be called, "The Malam Self-acting Tower Washer." January 5

787 W. G. Strype, Wicklow. Improvements in the treatment of blood to prepare it for use as a manure, or for other purposes. January 5

816 Dr. Les Gans, Frankfort-on-Main, and Dr. Meinhard Hoffmann, Mainkur, Germany. The manufacture of new colour-yielding products, and of colouring matter therefrom. January 5

832 A. J. Shilton, Birmingham. A new disinfectant, called "Affinitan." January 7

879 E. Loeflund, Stuttgart, Germany. An improved process for preserving milk or cream. January 7

890 J. Dearnley Wilson, A. Ambler, and J. Charnock Marshall, Sowerby Bridge. An improved process or means for extracting tannin or tannic acid from mirabalum nuts and other vegetable products. January 7

895 W. Beatson, Rotherham. An improved method of obtaining tin from tin scrap. January 7

903 J. Inray, London. An improvement in the manufacture of sulfoconjugate violet dyes. Communicated by La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, and Messrs. Poirier and A. Rosenstiehl, all of Paris. Complete specification. January 7

961 W. Martin, Milford Haven. An improved process for the manufacture of ammoniacal phosphates or phosphites. January 8

981 H. J. Haddon, London. A new or improved manufacture of binitro-hydrocellulose, and of explosives containing the same. Communicated by J. Hagemann, St. Anton, Tyrol. January 8

997 P. Barry, Tottenham. Improvements in iron manufacture. January 8

1024 E. Edwards, London. A new or improved explosive material to be used in blasting in mines and quarries. Communicated by L. Hensschen, Montjean, France. January 8

1031 W. P. Thompson, Liverpool. A new or improved colouring matter, and process for obtaining the same. Communicated by M. E. Savigny, New York. Complete specification. January 9

1032 W. P. Thompson, Liverpool. A new or improved colouring matter, and process for obtaining the same. Communicated by M. E. Savigny, New York. Complete specification. January 9

1074 J. P. Griess, Burton-on-Trent. Improvements in the manufacture or production of colouring matters from the tetrazo compound of benzidine-di-sulphonic acid. January 9

1079 G. Tidcombe, junior, Watford. Improvements in the

construction of strainers used for straining pulp in the manufacture of paper and paper-beards, and preparing "half-stuff" from wood, straw, and other materials for straining, clearing, or separating the shavings and other impurities from the same. January 9

1092 Major O. Glynn, Bolitho, Penzance. Improvements in the separation of ores and earthy substances. January 9

1099 J. P. Griess, Burton-on-Trent. Improvements in the manufacture or production of colouring matters obtained from sulpho-derivatives of benzidine, and in the preparation of such sulpho-derivatives. January 9

1129 R. Jardine Gibson, Dundee. Improvements in treating jute and other fibrous substances preparatory to dyeing the same. January 10

1198 H. Stier, Zwickau, Germany. Improvements in the treatment of carbonaceous substances in ovens or furnaces for the conversion thereof into combustible and other products. January 10

1223 T. Parker, Wolverhampton. Improvements in the production of yellow metal, partly applicable also to the founding of brass and bronze generally. January 11

1230 T. Ferneley Wiley, Hammersmith. Improvements in apparatus for enriching illuminating gas. January 11

1260 E. R. Southby, London. Improvements in treating grain for brewing and analogous purposes, and in apparatus therefor. January 11

1279 F. Hille, Chiswick. Improvements in treating sewage, and in disposing of the sludge by manufacturing artificial manure from the same. January 11

1294 C. Rennie Cowie, Glasgow. Improvements in the manufacture of artificial stone. Complete specification. January 12

1308 G. Hitchen, Darwen. Improvements in calcining furnaces for recovering soda ash. January 12

1340 R. Smith Casson, Brierley Hill. Improvements in the treatment of coke and peat. January 12

1341 S. Gilchrist Thomas, London. Improvements in the manufacture of phosphates. January 12

1342 S. Gilchrist Thomas, London. Improvements in the manufacture of alkalies and phosphates. January 12

1343 S. Gilchrist Thomas, London. Improvements in the manufacture of alkaline silicates and other alkaline salts. January 12

1350 F. McNamee, Liverpool. Improvements in the manufacture of spirits or alcohol, and of manure from certain waste materials. January 11

1412 L. Roth, Wetzlar, Germany. An improved manufacture of cement. Complete specification. January 14

## FRANCE.

1883.

154902 Vorster and Gruneberg. A process for removing ammonia from lighting and other gas, and obtaining sulphate and other salts of ammonia thereby. April 16

154920 Armour. Treatment of the residues or waste of collieries, of peat, and other coaly substances. April 17

154921 Appert Brothers. Moulding glass and dividing cast glass into various shapes and sizes. April 17

154927 Charmes, Montpelier. A tubular perforator for employing sulphide of carbon. April 17

154928 Schneider. A liquid for destroying the phylloxera. April 18

154930 Fancheux, Loos. Obtaining metallic bases or oxides. April 18

154935 Stier. Kilns for obtaining coke, and for distilling purposes. April 18

154939 Dannenberg. A kiln with a gas-chamber and movable sole for burning bricks, pottery, and calcining lime, cement, and strontianite. April 18

154947 Hulin. Processes and apparatus for the simultaneous production of coke and combustible gas. April 18

154952 Gandel and Mrs. Flaux. Applying Bunsen burners of sulphate of baryta, strontian, and other infusible substances to incandescent lamps. April 18

154979 Bang and Sanguinetti. A diffusion process for extracting oil and fat bodies. April 19

154980 Canet, Toulouse. Apparatus for fermenting alcoholic beverages. April 17

154991 Breyer. Obtaining micro-membranes for removing microscopic organisms from liquids and gases. April 20

154997 The Pancalastite Company. A single or double-acting explosive, with a base of bitumen or tar. April 18

155006 Stocker. An incombustible cellulose-plastic product. April 20

155014 Blitz. Obtaining a kind of fibrous, ligneous, white or coloured cellulose resembling rag pulp, etc. April 20

155026 Pictet and Brelaz. Obtaining chemical wood pulp. April 21

155032 Hyam and Howell. Processes and apparatus for tanning hides, leather, etc. April 21

155034 Dittler and Company. Obtaining yellow and red colouring substances. April 21

155048 Delaine, Fossex, Extracting beet-juice. April 25

155053 Sezille, Leballois-Perret. Obtaining maize yeast by acid saccharification. April 23

155058 Jameson. Dry distillation. April 23

155070 Mignon and Reuart. Apparatus for the production of cold. April 21

155080 Minguet and Jolibois, Paris. A liquid atomiser for distillation. April 21



155085 Weiler and Company. Separating paratoluidine from orthotoluidine, and also aniline and paratoluidine, by means of phosphates and arseniates. April 24  
 155091 Clauond. Obtaining hydrogen. April 24  
 155098 The Nickel and White Metal Foundry. Manufacturing pure and malleable nickel and cobalt and their alloys with iron. April 21  
 155107 Young and Beilby. Treatment of coal and other substances for obtaining ammonia or ammoniacal compounds, and heating gas; also apparatus belonging thereto. April 25  
 155108 Marchand. Obtaining binoxides of barium and calcium. April 25  
 155113 Deinhardt. Boiling hops. April 25  
 155116 Laguarigue De Survilleir, St. Pierre. An electric sifter for extracting the magnetic oxidulated iron ore  $\text{Fe}_2\text{O}_3$ , discovered in the ferruginous sand of the isle of Martinique and other colonies. March 21  
 155127 Gibson, junior, and Platt. An apparatus for soaping or ungreasing and dyeing tissues. April 26  
 155151 Julien, Castres. A mechanical process for extracting hurdocks, grain, straw, and other vegetable remains from textiles. April 20  
 155158 Goirand. A solution of camphor in alcohol for destroying the phylloxera. April 28  
 155174 Krupp. Obtaining compound plates and other products by welding hard iron and steel on soft iron and steel. April 30  
 155181 Spielhagen. Soap making. April 30.

#### Certificates of Addition.

154144 Possoz. Extracting sugar from molasses, etc. April 17  
 150936 Lissagaray and Leplay. Extracting tallow for obtaining oleo-margarine. April 18  
 138811 Richerol. A mixture called "alunoxium," for the manufacture of pipes, pottery, etc. April 19  
 148958 Scheibler. Separating sugar from molasses and syrup by production of saccharate of strontia. April 21  
 151671 Jameson. Obtaining coke. April 24  
 132179 David. An economical production of active oxygen and peroxide of hydrogen. April 30  
 150835 Dubus, Coget, and Company. Extracting fat, gelatinous, or gummy substances from tissues, etc. April 26

#### UNITED STATES.

289543 I. Levinstein, Manchester, England. The manufacture of yellow colouring matter. April 5, 1883  
 289545 Dugald McEchran, Greenock, Scotland. An apparatus for separating iron from charcoal, etc. December 20, 1881  
 289587 E. Warne, Easton, Pa. A method of and apparatus for reducing and disintegrating ores. February 25, 1882  
 289588 A. J. Weeks and J. E. Weeks, Littleton, N.H. A process of tanning. September 11, 1883  
 289613 O. Bredt, Barmen, Germany. The manufacture of red colouring matter. June 12, 1882  
 289706 J. E. Pontefract, Pittsburg, Pa. A process of and apparatus for distillation. October 15, 1883  
 289717 E. Sanser and M. Haller, Cincinnati, Ohio. A device for cooling beer. June 15, 1883  
 289755 S. R. Divine, Loch Sheldrake, N.Y. A process of preparing explosive compounds. July 23, 1883  
 289756 S. R. Divine, Loch Sheldrake. An explosive compound. July 23, 1883

289757 S. R. Divine, Loch Sheldrake. An explosive compound. July 23, 1883  
 289758 S. R. Divine, Loch Sheldrake. An explosive compound. July 23, 1883  
 289759 S. R. Divine, Loch Sheldrake. An explosive compound. July 23, 1883  
 289760 S. R. Divine, Loch Sheldrake. An explosive compound. August 1, 1883  
 289761 S. R. Divine, Loch Sheldrake, and A. C. Rand, New York. A method of preparing blasting cartridges. August 22, 1883  
 289762 S. R. Divine, Loch Sheldrake. An explosive compound. August 8, 1883  
 289763 S. R. Divine, Loch Sheldrake. An explosive compound. August 8, 1883  
 289764 S. R. Divine, Loch Sheldrake. A blasting cartridge. August 7, 1883  
 289765 S. R. Divine, Loch Sheldrake. A composition for preparing explosive compounds. August 7, 1883  
 289781 E. N. Riotte, New York. A process of obtaining precious metals from ores by amalgamation. July 13, 1882  
 289785 J. Stuart, Traer, Iowa. An evaporating apparatus. October 17, 1883  
 289788 H. Ujhely, Vienna. A process of decolorising and deodorising heavy mineral oils. February 24, 1883  
 289791 Adam Wingard, San Francisco, Cal. A dry-ore pulveriser and concentrator. September 16, 1882  
 289836 J. B. Houson, San Francisco. A process of and apparatus for obtaining boracic acid from native borate of lime. January 19, 1883  
 289842 E. J. Jerzmanowski, New York. Apparatus for producing gas. April 3, 1883  
 289887 J. Butler, Kennon, Ohio. A coke-oven. June 13, 1883  
 289914 E. J. Jerzmanowski, New York. A process of producing combustible gas. March 27, 1883  
 289939 J. Stickle, Denver, Colorado. A process of moulding and treating slag for paving and other purposes. March 23, 1883  
 289979 C. D. Chase, St. Louis, Mo. An apparatus for extracting gluten from the waste water of starch works. September 8, 1882  
 290016 T. D. Fairfield, Cleveland, Ohio. A process of and apparatus for chilling paraffin oils, etc. Nov. 9, 1883  
 290075 C. Leveque, Pouzin, France. A portable refining and oxidising apparatus. February 13, 1883  
 290079 J. Longmore, Liverpool, Eng. The manufacture of soap and extractive matter from cotton-seed, etc. October 5, 1883  
 290083 A. P. Massey, Cleveland, Ohio. An apparatus for use in the extraction of oils from seeds, etc. November 5, 1883  
 290105 Narcisse Pigeon, Yonkers. Evaporating liquids, etc. September 26, 1883  
 290110 E. Raw, Hartford, Conn. An apparatus for dyeing with hydrocarbons. May 25, 1883  
 290115 B. O. Rusfel, Denver, Col. A roasting and smelting furnace. August 20, 1883  
 290133 J. Stanley, New York. A metallic plastering surface. October 16, 1883

#### Reissues.

10415 J. B. Jones, Brooklyn. An alloy for coating metals. Original No. 23482, November 16, 1880.  
 10419 C. C. Gilman, Eldora, Iowa. A fireproof composition. Original No. 218091, October 11, 1881  
 10420 C. C. Gilman, Eldora. A fireproof composition. Original No. 218091, October 11, 1881.

# THE JOURNAL

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## A MONTHLY RECORD

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### NOTICES.

#### ANNUAL GENERAL MEETING.

The next Annual General Meeting of the Members of the Society of Chemical Industry will be held at Newcastle-on-Tyne, in July.

It is proposed that at this meeting, after the transaction

of the ordinary business of the Society, and an address from the president, a discussion shall take place on all the more important of the proposed methods for the utilisation of the condensable products obtainable from coal used in the manufacture of coke, coal used in blast furnaces, and coal used as fuel. Members who desire to read papers on any branch of this subject are requested to communicate with the general secretary. Each paper should be of such length that its reading shall not occupy more than twenty minutes.

It is intended that the members attending the meeting shall have an opportunity of dining together, and it is expected that they will be invited to visit various important chemical and other works.

At a meeting of the Council of this Society, held on 21st December, 1883, it was resolved:—

1. That after December, 1883, the price of the Society's Journal to non-members be 30s. per annum, and that an announcement to that effect be printed on the cover of the Journal.

2. That the price of sets or annual volumes of the Journal to members of this Society be 21s.

3. That the price of single copies of the Journal be 2s. 6d. each to members only, and that no single copies be sold to the outside public at all.

4. That due notice being given by the Secretary, the issue of the Journal shall cease to those members who have not paid their subscriptions for 1883, commencing with the number for January, 1884.

5. That, due notice being given by the Secretary, those gentlemen who have failed to pay their subscriptions for 1882 and 1883 shall cease to be members of this Society after January 21st, 1884.

Members are requested to notify change of address as promptly as possible to the General Secretary and to the Honorary Secretary of the Section to which they belong in order to insure receipt of the Society's journal.

Members will please to take notice that the Annual Subscription of £1 1s. for 1884 is now due, and should be forwarded to the Hon. Treasurer, to whom also cheques and orders should be made payable.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the first volume of the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

Numerous inquiries have been made for complete sets of the Journal for 1882. Some of the numbers for that year are out of print. Members who have not the volume for 1882, and wish to obtain it, are invited to signify that wish to the Secretary as early as possible. If a sufficient number of applications for the complete set are received, the missing numbers will be reprinted.



## LIST OF MEMBERS ELECTED FEBRUARY 22nd, 1884.

James Dunnachie, Glenboig, near Coatbridge, N.B., fire-clay manufacturer.  
 James Middleton, 179, West George Street, Glasgow.  
 Alexander Crum, M.P., Kelburne, Fairlie, Greenock, calico printer.  
 J. B. Hutchinson, University of Glasgow, chemist.  
 Captain H. A. Gadsden, 19, Australian Avenue, London, E.C., merchant.  
 Alexander Whitelaw, 87, Sidney Street, Glasgow, soap manufacturer.  
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 John Addie, Langloan Ironworks, Coatbridge, N.B., iron-master.  
 H. Pickus, Prussiate Works, Droylsden, Manchester, prussiate manufacturer.  
 H. M. Hastings, 61, Edith Road, West Kensington, W., analyst.  
 Edward Sykes, 10, Hill Street, Seedley, Pendleton, Lancashire, chemist.  
 John Blair, Irvine Bank Chemical Works, Kilmarnock, N.B., manufacturing chemist.  
 J. B. Binks, 18, Esk Street, Silloth, Cumberland, analyst.  
 W. Elborne, the Owens College, Manchester, lecturer on Pharmacy and Materia Medica.  
 Dr. R. Parkinson, Sun Bridge Buildings, Bradford, Yorkshire, pharmaceutical chemist.  
 William Crowther, Field House, Gomersal, Leeds, manufacturing chemist.  
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 F. Burton, 2, Green Street, Bethnal Green, E., manufacturer of dyestuffs.  
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 F. Oliver, 70, Winchester Street, South Shields, analyst.  
 Jas. Kenn, 47, Croft Terrace, Jarrow-on-Tyne, assistant manager of rolling mills.  
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 R. H. Bow, 7, South Gray Street, Edinburgh, civil engineer.  
 J. Moffatt-Johnston, Young's Paraffin Company Limited, Bathgate, N.B., chemist.  
 Shackleton Hallett, 1, Hare Court, Temple, E. C., barrister at-law.  
 U. Jeanneret-Grosjean, 97, Euston Road, London, N.W.  
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 Prosper Flanrez, 9, Rue Morris, a Bruxelles, chemical engineer.  
 Alfred Solvay, Boitsfort, Belgium, chemical manufacturer.  
 Louis Smet, 217, Chaussee de Vleurgat, Brussels, chemical manufacturer.  
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 Dugald Clark, Crown Ironworks, N. Woodside Road, Glasgow, engineer.  
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 Dr. Edmund Ronalds, Bonnington House, Edinburgh.  
 Robert Orr, Linenhatch Chemical Works, Falkirk, N.B., manufacturing chemist.  
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 Charles M. King, Campsie Alum Works, Lennoxton, N.B., alum manufacturer.  
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 A. Learmouth Adam, 18, Robertson Street, Greenock, N.B., analyst.  
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 B. H. Remmers, 189, St. Vincent Street, Glasgow, chemical engineer.  
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 C. M. Blades, Stanley Mount, Northwich, Cheshire, analyst.  
 Thos. Holgate, 133, Manchester Road, Burnley, gas engineer.  
 W. A. Bryson, 20, Lomond Road, Trinity, Edinburgh, chemical engineer.  
 Saml. Rideal, University College, London, W.C., chemist.  
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 F. W. Renaut, The Cottage, Burstow-by-Crawley, Surrey, secretary.  
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 Wm. Tebb, 7, Albert Road, Gloucester Gate, Regent's Park, N.W.  
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 J. R. Bennie, Kinning Park Foundry, Glasgow, chemical engineer.  
 W. J. A. Donald, 2, Belgrave Terrace, Glasgow, chemical manufacturer.  
 R. A. Inglis, Arden, Bothwell, N.B., chemist.  
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 C. Maenab, Lillyburn, Milton of Campsie, N.B., calico printer.  
 Jno. Miller, 154, Gunport Street, Glasgow, metallurgist.  
 G. D. H. Mitchell, Oakbank, Midcalder, N.B., chemist, oilworks.  
 Jas. Murrie, Kinning Park Foundry, Glasgow, civil engineer.  
 W. Paterson, jun., 8, Claremont Terrace, Glasgow, merchant.  
 Jas. Stevenson, 27, St. Vincent Place, Glasgow, manufacturing chemist.  
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 N. H. J. Miller, 8, Oppidans Road, N.W., assistant in Research laboratory.  
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 John West, Barfield House, Didsbury, near Manchester, civil engineer.  
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 J. E. Marley, Messrs. Sproat, Marley and Company, Hebburn-on-Tyne, ironfounder.  
 Thomas Kirkham, Runcorn, Cheshire, chemical contractor.  
 W. H. Michael, Q.C., 1, King's Bench Walk, Temple, E.C., barrister-at-law.  
 Thomas Akitt, 3, Victoria Road, Lenzie, N.B., chemist.  
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 James Duncan, Cleland, Lanarkshire, medical practitioner.  
 Henry Gall, chez MM. Pechiney et Cie, Salindres (Gard), France, chemist.  
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 W. M. Miller, 40, Whitehill Street, Dennistoun, Glasgow, analyst.  
 George S. Packer, Hallside Steelworks, Newton by Glasgow, manager.  
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 George T. Powell, Rotherwood, Sydenham Hill, S.E., solicitor.  
 James Riley, 150, Hope Street, Glasgow, steel manufacturer.  
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The meetings of this Section are held on the first Monday in each month.

On March 3, the following Papers will be read—(1) "On the Manufacture of Cuprammonium and Zincammonium, and their Technical Adaptation." (2) "On the Filtration of Potable Waters." (3) "On Some Applications of Kieselsäure."

CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE,  
Monday, February 4th, 1884.

MR. DAVID HOWARD IN THE CHAIR.

## ON THE DISPOSAL OF SEWAGE SLUDGE.

BY CHRISTOPHER CLARKE HUTCHINSON.

AMONG the many problems engaging the attention of sanitary engineers, that which embraces the removal and disposal of the refuse matters of a community, termed sewage, stands perhaps the most prominent. Ignorance of the principles of sanitary requirements, and of the many factors which contribute to the growth of disease resulting from the infringement of Nature's laws, is quite sufficient to account for the laxity of ideas which, even up to recent times, prevailed on this subject. But with the rapid advances of natural science those principles which are now recognised as governing health and disease were gradually developed, and we became alive to the fact that if we would be free from many forms of disease, it was essential to remove from our midst those materials which were either their direct cause or agents of transmission. It is curious to note, on a perusal of the many patented inventions bearing upon this subject, that up to even a very recent date inventors seemed to have in view the realisation of commercial profit, by the extraction of substances valuable for agricultural and other purposes, rather than the abatement of nuisance and the promotion of public cleanliness; but as communities grew, and the fallacies of such ideas were shown by the repeated failures of innumerable golden projects, many of which had for their object a goal somewhat commensurate with "the philosopher's stone" or "the elixir of life," it became generally recognised that the sewage question simply resolved itself into the disposal of the refuse and filth of our towns in the most economical and effectual manner. This is cogently expressed in the conclusions arrived at by the Committee of the Society of Arts\*: "For health's sake, without consideration of commercial profit, sewage and excreta must be got rid of at any cost." The question therefore resolves itself into disposing of this offensive matter in a manner best suited to the locality, having regard (1) to the sanitary efficiency of the means employed, (2) the economical attainment of the result, and (3) the reduction of enforced expenditure by the employment of measures of disposal affording some return of the means expended.

Prior to the passage of the Rivers Pollution Act in 1876, it was not incumbent upon every town, as specifically enforced by law, to abstain from turning direct into the nearest watercourse, without taking measures for purification, the whole of its sewage, and the onus of enforcing abatement of wholesale pollution rested with the riparian proprietor whose property might suffer deterioration. Such a means of disposal of course fulfilled no one of the above conditions; and it is only now tolerated in the case of those towns discharging their sewage into a tidal river, when it is assumed the impurities will be carried direct into the sea. How far such an assumption can be borne out may here be briefly alluded to in the case of the Thames. The metropolis is the only town on the Thames now permitted to discharge its sewage directly into the river without adopting "the best practical and available means" for its purification. The dry weather volume of sewage so discharged daily into the river, from the outfalls at Barking and Crossness, is upwards

of 125 millions of gallons, containing in suspension about 250 and in solution about 450 tons of solid matter. Now a portion of the 450 tons of matter in solution is capable of being precipitated, and doubtless by the action of oxidation as well as of actual precipitation by the fresh water of the river some portion is actually so thrown down. Consequently, it appears that from 250 to 300 tons of objectionable and putrescent solid matter, equalling about 3,000 tons of what is termed "sewage sludge," capable of depositing, are daily admitted into the river. Now if this could be carried away directly to the sea by the action of the tide, such a method of sewage disposal might—under the circumstances—not be held to be objectionable; but when we consider that the tidal outfalls are a distance of some 41 miles from the mouth of the river, it is apparent that the time occupied in reaching the sea must be considerable. It has recently been clearly shown by Professor W. Cawthorne Unwin,\* that if the travel of matter in the river were due to the upland water alone discharged into the river at Teddington, the passage of solid matter entering the Thames at Woolwich down to Sheerness would occupy about 65 days. This, he shows, is considerably modified by tidal action, but that viewed under the most favourable circumstances, the period of oscillation of a segment of sewage-laden water between the mouth of the river at Sheerness and the outfalls of the sewers is about 44 tides or 22 days. The effect of the presence of so large a volume of putrescent matter in the lower part of the river, placed under conditions favourable for deposition, can hardly be over-estimated. The clothing of the river banks with sewage mud, as well as the shoals of gelatinous sewage matter accumulating in the bed of the river, is in itself a convincing proof of the inefficiency of the disposal of the metropolitan sewage by the present tidal outfalls. How long this problem, the solution of which is not by any means overwhelming, will be permitted to rest unattacked may possibly only be determined by the occurrence in the future of an epidemic decimation, giving the necessary stimulus to more advanced metropolitan sanitary legislation, thereby removing what now exists as a blot upon the cleanliness of the largest and richest city of the world. Before entering into the details of the specific method for the abatement of the sewage difficulty now brought before your notice, it will be necessary to briefly touch upon the methods for sewage disposal now usually employed, so as to be better able to show the extent and importance of its bearing upon the whole question, irrespective of the definite system pursued.

Processes for the abatement of river pollution by sewage may be broadly grouped under two heads: (1) Processes of prevention. (2) Processes of purification.

### I.—PROCESSES OF PREVENTION.

Under this head are embraced all the various methods and contrivances for dealing with human excreta upon a distinct and separate system other than water carriage. But as this forms only a fractional part of the many elements of pollution, the alleviation it affords is only partial, while at the same time the system of water carriage has still to be resorted to. Hence it follows that, providing only for one portion of the general problem, it cannot in any case obviate a water-carried system and consequently measures of purification. How small is its preventive effect, as regards the polluting character of the remaining sewage, is shown by the table given

\* Report of the results of the Conference of Health and Sewage of Towns, 1876.

\* "On the Movement of the Water in the Tidal River with Reference to the Position of the Sewer Outfalls." By W. Cawthorne Unwin, M.I.C.E.



below, the figures in which are taken from the report of the Rivers Pollution Commission on the Mersey and Ribble basins.

AVERAGE COMPOSITION OF SEWAGE IN PARTS PER 100,000.

Description.	Solids in solution.	Organic carbon.	Organic nitrogen.	Ammonia.	Nitrogen as nitrates.	Total combined nitrogen.	Chlorine.	SUSPENDED MATTERS.		
								Mineral.	Organic.	Total.
Water-closet Towns..	72.2	4.696	2.205	6.703	.003	7.728	10.66	24.18	20.51	44.69
Midden, &c Towns..	82.4	4.181	1.975	5.435	.000	6.451	11.54	17.81	21.30	39.11

Now without entering into details of the expenditure incident to the various systems included under this head, it will be at once apparent that as the means employed do not sensibly affect the question of alleviation, it follows that the expenditure upon the process of purification, which must be employed upon the bulk of the sewage, cannot be very much (if at all) diminished, consequently such measures do not satisfy the first two of the requirements before set forth, viz.,

Years of experience under varied conditions have, however, demonstrated this process to be not altogether so free from difficulty, or so remunerative as was anticipated. In the first instance suitable land is not always procurable, then again the area required, so that the requirements of the vegetation may be considered, as well as the cleansing of the sewage, becomes enormous. From 25 to 50 persons per acre of land has been estimated, from results obtained by experience, as the correct basis of operation. Such proportions would necessitate the employment of enormous areas to be brought under irrigation culture, a few instances of which are given below:—

Town.	Population.	Area required at 1 acre=50 persons.
Leeds .....	250,000 .....	5,000
Manchester .....	360,000 .....	7,200
Glasgow .....	540,000 .....	10,800
London .....	3,500,000 .....	70,000

Nor is this alone the only difficulty. It has become from experience a well-known fact that the purifying effect of an area of land continuously used for purposes of irrigation is not lasting, and that ultimately the soil becomes so saturated that a serious nuisance, both disagreeable to the senses and injurious to health, is the result. On this head the experience of Mr. Hawksley may be quoted\*: "Water irrigation carried

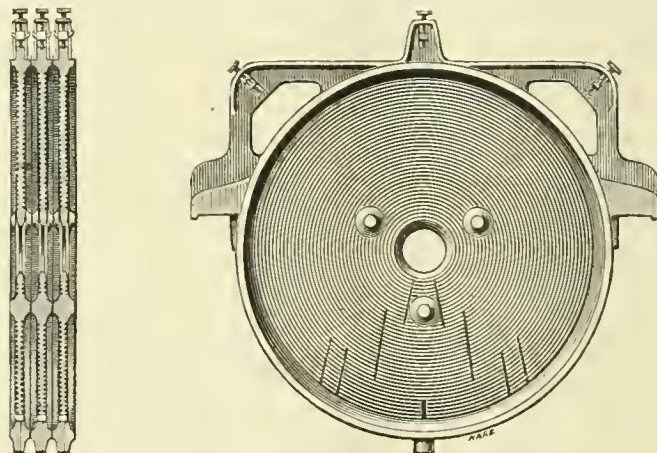


FIG. 1.

(1) efficiency, (2) economy. How far it meets the remaining and minor consideration will be noticed after on.

#### II.—PROCESSES OF PURIFICATION.

The means employed for the purification of water-carried sewage are all to be classed under three heads: (a) Irrigation. (b) Intermittent filtration. (c) Chemical precipitation. There are, however, instances, more particularly in later practice, and in cases which altogether depend upon the local conditions and required standard of purification, in which no one of these methods is pursued in its entirety, but an attempt is made to produce the most efficient and economical result by combining the salient advantages of two or more. It will, however, perhaps be more convenient to notice each in its entirety, so that the drawbacks and difficulties attendant may be shown.

(a)—IRRIGATION.—At first sight it would appear that under all circumstances where it was possible to procure the necessary area of land, the method of purification by broad irrigation, attaining at the first glance the double object of extreme purification and the realisation of the "mine of wealth" sewage has always been understood to contain, would be the most rational course to adopt.

on in warm weather is exceedingly unhealthy. . . . I can speak positively to it from repeated observation in different places, that the odour, particularly at night, and particularly upon damp evenings in autumn, is very sickly indeed, and that in all these cases a great deal of disease prevails. The sewage forms a deposit on the surface of the ground, that deposit forms a cake of organic matter, and that organic matter when it is in a damp state, as it usually is, gives off in warm weather a most odious stench." Evidence of a similar nature could be multiplied to almost any extent, all of which points to the conclusion that if irrigation is to be practised with any measure of success from a sanitary point of view, it becomes necessary to adopt measures for removing the putrescent solid suspended matter, either by subsidence or partial chemical treatment, in order to avoid the clogging of the pores of the ground and the formation of a film of putrefactive matter injurious alike to the health of the neighbourhood and the vegetation of the ground itself. Such being the case, it is evident measures must be adopted for the effectual disposal of the resulting accumulations of sludge by the most efficient and economical process which

\* Mr. Thomas Hawksley, C.E., evidence before committees of the House of Commons. 1876.



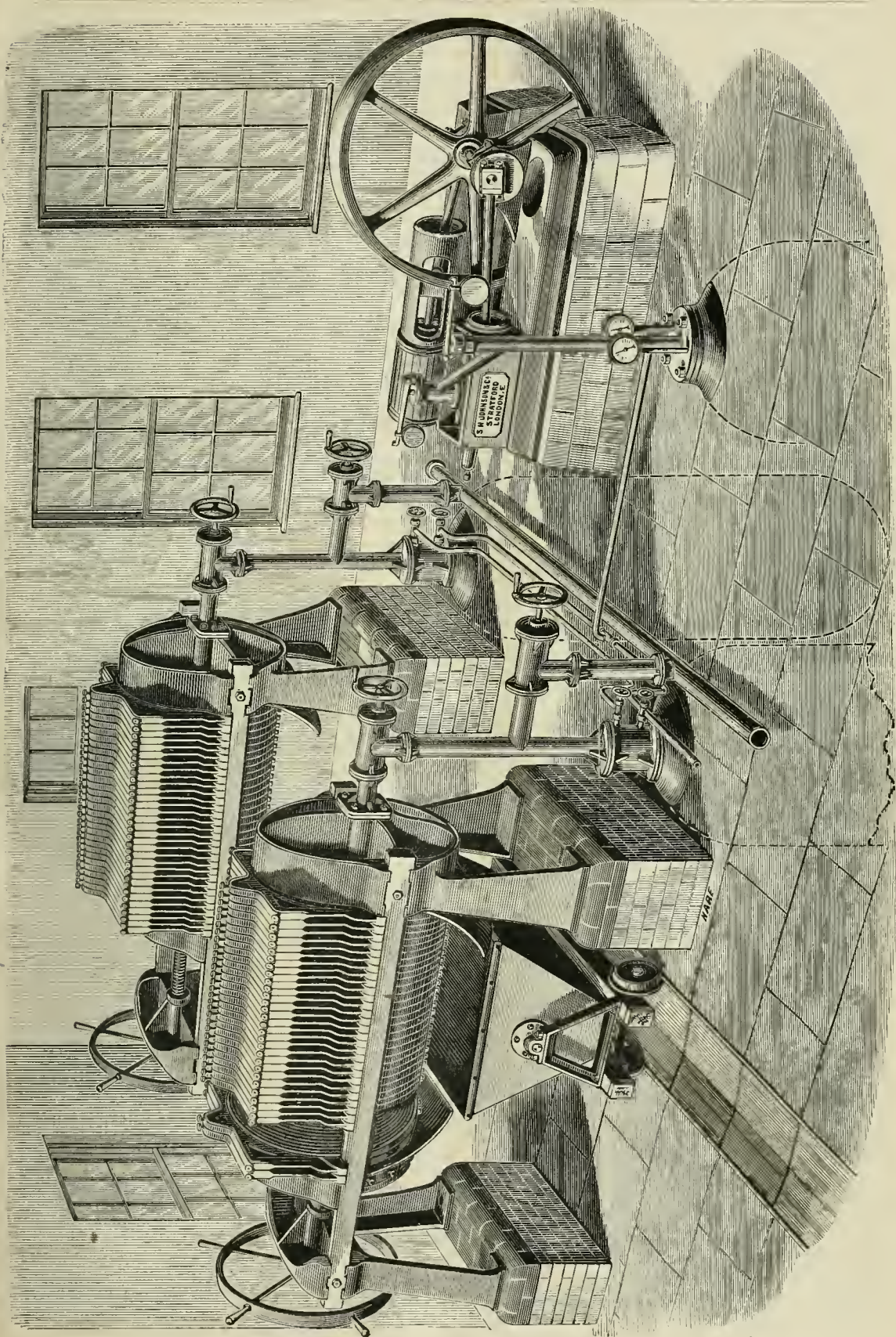


FIG. 2



can be employed. Both in this and the two succeeding cases such means of disposal will be considered separately. Viewed from an economical standpoint, it will be amply sufficient for the purposes of this paper to state that experience has shown that even where the local conditions are favourable no great return can be expected. "Great hopes were a few years ago entertained that in irrigation had been found the grand solution of the sewage question.

All that is changed now. Sewage farms on which immense sums of money have been expended have been reluctantly abandoned, and irrigation is no longer regarded as anything more than a means of obtaining a good effluent at a moderate outlay.\*

(b) **INTERMITTENT FILTRATION.**—The experiments of Dr. Frankland, published in the First Report of the Rivers Pollution Commissioners of 1868, clearly show that the passage of sewage water *through* a properly prepared filtration area of soil effects a purification far in excess of the effect which would be produced by irrigation *over the surface* of the same area. It is further shown that it is possible, by means of an acre of properly drained and prepared soil, six feet deep, *used intermittently*, to effect the purification of the sewage resulting from a population of 3,300 persons, sufficient to enable the water to be used again for all but domestic purposes, the result of such treatment being that, by means of oxidation, nearly the whole of the organic matter in *solution* is transformed into innocuous inorganic salts. It is, however, manifestly essential that as the continued efficiency of the same area depends entirely upon its thorough aeration during intervals of rest, it should be kept in such a condition that it can readily absorb a fresh supply of oxygen to bring to bear upon a further quantity of sewage. It therefore follows that to maintain the porosity of the soil it is essential to keep the suspended decaying organic matter contained in the sewage from blocking up the pores of the soil. This can only be effected by its interception; and as a consequence, as is the case in irrigation, whatever be the means employed there will be the inevitable accompaniment of sludge accumulation. In the report before referred to this is pointed out as follows: "It is not unlikely that the operation, involving as it does the exposure of large and offensive filtering beds to the air, would be itself attended with much serious nuisance. The previous treatment of the sewage by one of the chemical processes already described would, however, obviate this liability to nuisance, whilst it would probably reduce by one-half the size of the filter necessary for cleansing a given volume of sewage. Such a combination of a chemical method with intermittent filtration offers the most hopeful process." It therefore appears that this method, like irrigation, cannot be efficiently used without some means for the disposal of the sludge. Several applications of this process could be alluded to—for instance, the works of the Croydon Rural Sanitary Authority, designed by Mr. Baldwin Latham, in which case the filtration area is so disposed, both in size and treatment, as to be brought under cultivation as a sewage farm, and thus return some portion of the expenditure.

(c) **CHEMICAL PRECIPITATION.**—In all those situations where neither of the foregoing methods can be used, or where so high a standard of purification is not essential—such as, for instance, discharge of the effluent into a tidal river, or a stream of such a size that, compared with the volume of the effluent, contamination is reduced to a minimum—chemical treatment can be most advantageously used. We have

likewise seen that where a high standard of purity is desirable the process offering the best hopes of success is that of chemical precipitation succeeded by intermittent downward filtration through a small area of land. It has been over and over again raised as an objection to chemical precipitation that, no matter what special reagent is used, the ultimate difficulty is the same, namely, the disposal of the large accumulations of sludge. In a paper read before the Institution of Civil Engineers by Mr. C. Norman Bazalgette,\* the author stated, as objection to chemical precipitation, that "it is impossible to manipulate the enormous accumulations of sludge necessarily incident to treatment by chemicals," and further, that when chemical treatment preceded natural filtration, "the accumulation of sludge is an objection inseparable from their use," *i.e.*, chemicals. It has, however, been before shown that irrespective of the method of purification pursued, the same ultimate difficulty results, *viz.*, the formation and accumulation of sludge. So that, instead of the evil resulting from chemical processes as such, the sludge difficulty is one inseparable from efficient modes of sewage disposal. Such being the case, the importance of an economical and efficient mode of dealing with the sludge can hardly be overestimated, because, to quote the words of Mr. Shelford,\* "information obtained showed distinctly that the sewage question resolved itself into the difficulty of disposing of the solid matters in suspension."

#### DISPOSAL OF SLUDGE.

Sewage sludge proper, as left after precipitation, is a thin mud, containing from 90 to 95 per cent. of water, the remaining part consisting of the solid matter, organic and mineral, originally suspended in the sewage, a small amount of the soluble organic matter precipitated by the chemical reagents, and more or less of the reagents themselves used for defecation. The large quantity of water it contains renders it exceedingly bulky, and this, together with its offensive character, constitutes the chief difficulty of its treatment. Unless deprived of the greater portion of its moisture, however precipitated or obtained from the sewage, it enters in putrefactive fermentation of a more or less violent and offensive nature. As obtained from the various chemical processes, it is yielded in quantity equivalent to from 1 to 1·25 ton for every thousand inhabitants contributing to the sewage; but this, together with its character, depends upon the nature of the district, whether manufacturing or rural, the time of year, the mode in which it is obtained, and a variety of other circumstances. No material, perhaps, varies more in its character than sewage sludge, unless it be the sewage from which it is collected. Many attempts have been made to reduce it to a condition in which it can be handled and got rid of, the majority of which have directly had for their object the recovery of the small amount of manurial matter it contains. How little success or economy these various appliances have attained is attested by the almost universal resort had to the mode of dealing with the sludge up to very recently. This mode, primitive enough in its simplicity, consists in draining the accumulations of deposit into large pits, several feet deep, where it is allowed to remain until it has parted with a sufficient amount of moisture to allow it to be dug and carted away from employment as manure. Even after being thus exposed for a period of as much as two years, evaporation and soakage have removed only a comparatively small amount of its water. In the meantime, the offensive odours given off pollute

\* "Methods of Disposing of Sewage Adopted in Various Towns in England" (Report to the Town Council and Board of Police, Glasgow, 1878).

\* "Minutes of Proceedings of Civil Engineers," vol. xlviii.

the atmosphere of the whole neighbourhood, experience of which no doubt originated the remark quoted by Messrs. Rawlinson and Read,\* "that the nuisance chargeable to such works is due not to the act of precipitation but to the process employed for drying the solid matter." We may briefly summarise the methods attempted under two heads—(a) Evaporative methods. (b) Mechanical methods.

(a) **EVAPORATIVE METHODS.**—All such processes as depend upon the application of artificial heat cannot attain any degree of success, (1) on account of the expense they entail both for fuel and labour, (2) the pollution of the neighbourhood of the works by offensive odours inevitably given off. The removal of the water by evaporation, as will be apparent, is infinitely more complex than the simple evaporation of water in an ordinary boiler, in which, under ordinary circumstances, good fuel will evaporate from 7 to 9 times its own weight of water. So high an evaporative efficiency is impossible with sludge, and it is more than probable that not more than one-third such effect has ever been attained. Upon this basis of calculation the following shows the quantity of fuel required for reducing the sludge to a condition in which it can be easily handled, containing 50 per cent. of water. One ton of wet sludge contains—

Solid matter (40%) = 2 cwt.  
Water matter (60%) = 18 cwt.

A sludge yielded by drying down to 50 per cent. moisture contains—

Solid matter = 2 cwt.  
Water matter = 2 cwt.  
Water evaporated = 16 cwt.  
Coal required to evaporate 16 cwt. of water at efficiency given above } = 6 cwt. (about)

The cost for labour and maintenance is proportionately high likewise. The cost of drying by Milburn's machine, at one time in use in several sewage works, has been stated to be in one instance £1 per ton, and in another £1 8s. 9d. per ton. Such expenditure cannot, with a material of so little value, offer any hopes of success, and, accordingly, we find that machines of the evaporative type have been altogether abandoned.

(b) **MECHANICAL METHODS.**—Under this head we may perhaps first notice the method of disposing of the wet sludge by pumping it direct from the tanks on to land, into which, after air-drying, it is dug. At Birmingham the sludge produced by lime treatment, amounting to upwards of 350 tons per day, is got rid of in this way, and it was originally thought that it would so quickly rot away that within two years the same land could be used over again. Such, however, is not the case. In 1877 Mr. J. Mansergh, C.E., stated, "Such a process as that of digging in the immense quantity of sludge produced by the lime process at Birmingham was unlikely to be of long duration;" and his remarks have been fully borne out. Ninety acres of land are annually required for this purpose, and if continued much longer it cannot be denied the whole area must ultimately become a mass of sewage filth. In 1876 the cost of removing from the tanks and digging in 109,500 tons of sludge is returned as £12,778, equal to about 2s. 4d. per ton. Mr. Ed. Monson has proposed to deal with the sludge by making it into bricks, the manufacture so carried out effecting a return for the expenditure upon the purification. This plan seems to have been tried in one or two cases—for instance, Leicester and Birmingham—but from reasons sufficiently obvious I am not aware that it has ever been adopted. Brick-making, as already pursued, is an industry far from

being agreeable. What it would become when in addition there would be added those infinitely worse odours of first baking, and then slowly burning the organic matters contained in the sludge-made bricks, can perhaps be better imagined than described. The brick shown was made by this process from the sludge accumulated at Leicester, where there is sufficient bulk of old deposit alone to test the efficacy of the process for some years to come. Many other similar proposals for mechanically dealing with the sludge have at different times been made, amongst the most successful being the late General Scott's cement process; but as in each instance the bulk of the water must be eliminated before their object can be attained, consideration of them is beyond our present purpose. Separation of the liquid and solid matter has frequently been attempted by the use of centrifugal machines; but however admirably adapted to the expulsion of a mother-liquor from a crystalline magma, where the force of separation to be exerted is comparatively small, and the proportion between the solid and liquid fairly even, little effect is obtained by their use in materials of the same slimy character as sewage mud. Wicksteed patented their application to this purpose in 1851, and up to 1870 they appear to have been occasionally used. Not more than one-half of the water can be removed by them, and the residue, consequently, is not in a condition in which it can be easily handled, nor is its offensive character much modified. Such a result, which even then is only obtained by considerable expense, quite justifies the remark made in 1870 by Dr. Odling, when asked his opinion of the process: "It seemed to me to be perfectly inefficient." Of all modern industrial appliances for the separation of liquid from solid matter the class of apparatus known as filter presses offers the most practical advantages, being capable of such modifications as admit of their successful application to an infinite variety of purposes. As manufacturers of such apparatus the firm of which I am a member (S. H. Johnson and Company, of Stratford), with a knowledge of the many unsuccessful attempts made in the directions indicated in the foregoing part of this paper, as well as the ineffectual attempts to adapt the filter press also to the same purpose, began some five or six years since to turn their attention to the production of suitable and efficient machines. As may be imagined, such a system was not easily elaborated; but after some three or four years' attention to the many details necessary to be worked out practically the whole of the difficulties have disappeared, and we are now able to place before you machinery which for a considerable period has been satisfying the conditions of the problem we have been considering, and to a description of which we shall now pass. A filter press consists of a number of narrow cells held in a suitable frame, the interior faces being provided with appropriate drainage surfaces communicating with an outlet, and covered by a filtering medium, generally cloth or paper. The interior of the cells so built up are in communication directly with each other, or with a common channel for the introduction of the matter operated upon; and as nothing introduced into the cells can find an exit without passing through the cloth the solid matter fills up their interior, the liquid leaving by the drainage surfaces. The cells of the machine being subjected to an hydraulic pressure, which increases as the operation goes on, and as the growing resistance offered by the increasing thickness of solid matter on the cloth, must of necessity, on their exterior touching surfaces, be mechanically made true and pressed together with force sufficient to prevent the material operated on escaping. The force exerted upon the material

\* "Modes of Treating Town Sewage," 1876.



driven into the press, whether by a pump or other means, must be considerable; and as both sides of a cell are subjected to the same pressure in opposite directions, it follows that if nothing, such as a local stoppage, interferes with this equality of pressure, the diaphragms are in equilibrium as regards pressure. To be of any utility, in dealing with daily accumulations of sludge, the machines must be of considerable size, because, even for a population of 30,000, about 30 tons have to be dealt with during the day. Difficulty of making large plates sufficiently rigid and tight at once appears, but as far as this is concerned it has been met by adopting a circular shape, a form which above all others for strength and resistance to the internal fluid pressure is at once apparent to the mechanical

equilibrium of opposite pressures on each side of the adjacent cells is destroyed, and the plates, even under moderate pressure, unless made of a thickness quite impracticable, collapse and break, a result which the usual size of the machines, 3ft. and upwards in diameter, readily encourages. For example, at Coventry the employment of two such presses, each with 24 plates, led to the destruction of no less than upwards of 60 plates in a few weeks from no other cause. The remedy for this is as efficient as it appears simple. Instead of the central parts of the cells being kept entirely apart, suitably-shaped projections are formed on their surfaces, which are faced down to the same plane as the exterior joints, so that when the machine is

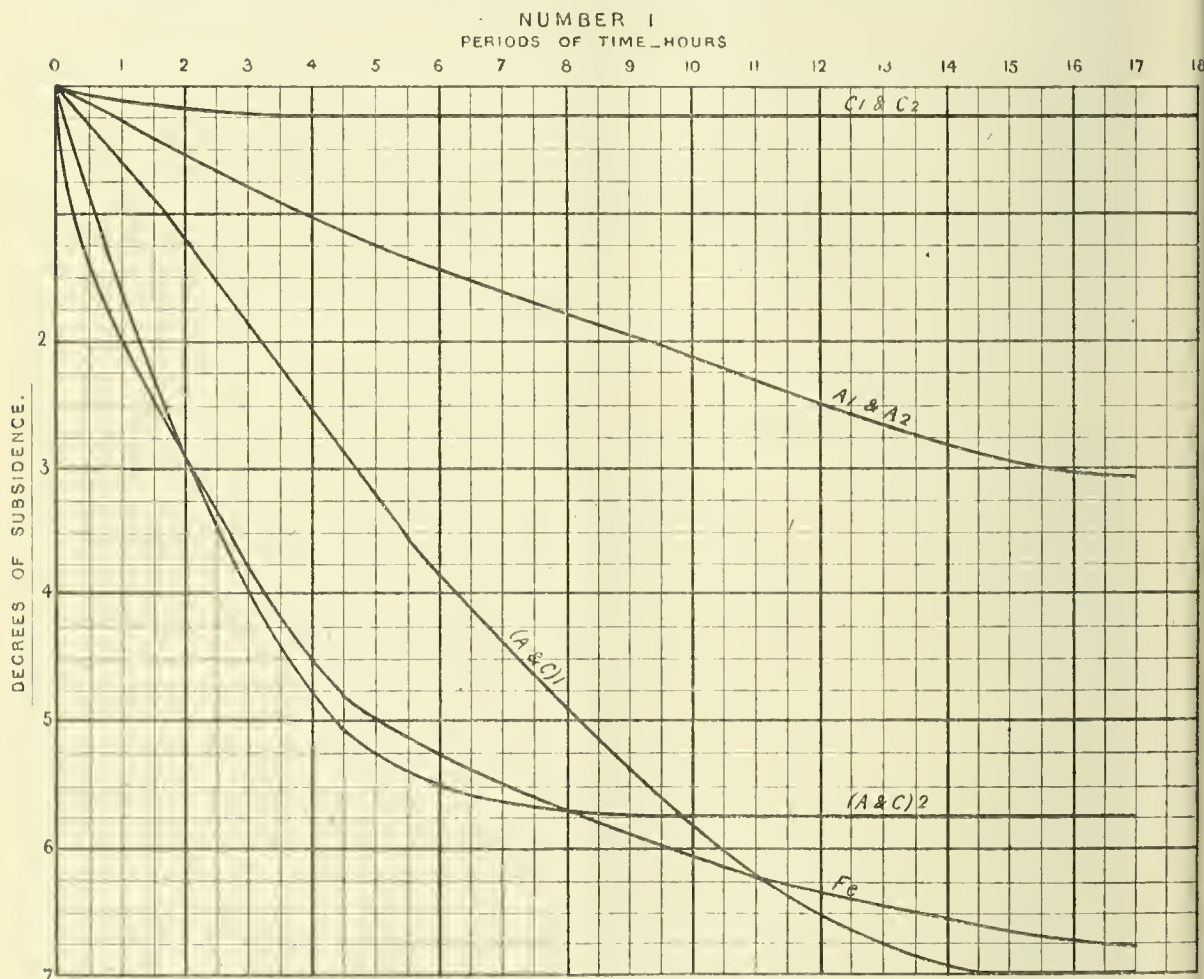


Fig 3

mind. It would be impossible to enter into all the minor details of construction which experience has proved to be necessary to success, but although not here noticed they are none the less important, as each in its particular manner contributes to the result. Only the leading and most vital features will therefore be dwelt upon. The most disastrous difficulty we have had to contend with has been the stoppage of the feed passages, due to the heterogeneous character of the rubbish and fibrous matter present, which only too easily builds itself up, whenever the least excuse offers, into hard blocks. The inevitable consequence of the stoppage of any of the chambers is that the

screwed up these nip the cloths between them, and bearing upon each other form a series of stays from one end of the machine to the other, supporting the plates when the equilibrated pressure is destroyed, until the higher pressure forces the obstruction away. Since the introduction of this simple device no fracture has occurred in any machines to which they have been applied, and experience enables us to state positively that without such a method of support no filter press can be successfully used for sewage purposes. This plan will be clearly understood from the woodcut (Fig. 1), showing a front view and section of large sewage filtering plates. Another requirement equally important is the mode of in-

troducing the sludge into the press. In most cases of filtration an hydraulic pump is used, and where the matter is not too thick, and contains *little or no fibrous matter*, answers sufficiently well with small quantities. But we speedily found that with the mass of fibrous matter and rubbish with which we had to deal the valves of the pumps continually became choked, and operations were inevitably interrupted at the most vital period. The large quantity of gritty silicious matter, as would be expected, rapidly cut the working parts to pieces. The plan has therefore been followed of running the sludge, or drawing it by *vacuo*, into cylinders placed underneath the presses, and large enough to contain a charge. From this vessel it is forced by compressed

speed of the compressor can be kept constant and the power stored up, ready to be drawn upon when required. Again, pumps must not be worked for such a purpose above about 40 strokes per minute: consequently the rate of feed at the commencement of the pressing is too slow. An efficient air compressor can be constructed to run at 100 strokes per minute, and therefore, irrespective of air accumulated, the press can be fed rapidly, economy of time being the result. Other important advantages could be enumerated. The addition of a small quantity of fresh lime just before pressing considerably adds to the facility of the operation. The results of the operation, briefly described, are—within one hour every five tons of wet sludge containing 90 per cent.

NUMBER II.  
PERIODS OF TIME—HOURS.

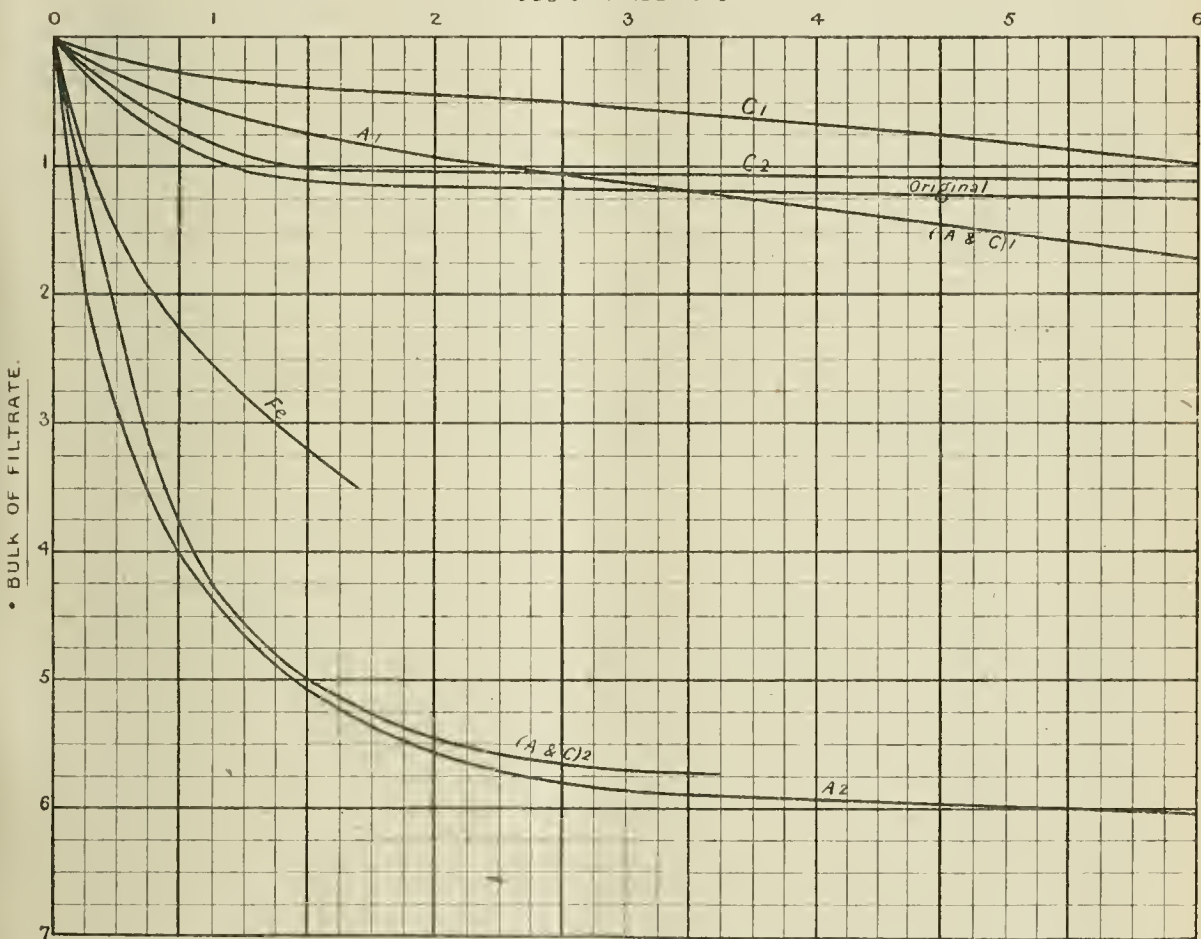


Fig 4

air, of a pressure of 100lb. to 120lb. per square inch, into the press, the air being supplied by a suitable compressor, pumping in the case of two or more presses into a storage vessel. The press is thus instantaneously filled, filtration commenced immediately, and the whole operation is completed in about one hour. The advantages of the compressed air system are many besides that which has been shown. It is found to be more economical than by employing pumps if the quantity of material to be operated upon is large. It is of course evident that as the filter press operation approaches completion less material is required. With a pump this would escape through the relief valve, and the power be lost; with compressed air, working on an elastic medium, the

of water can be deprived of 88 per cent. of its water, giving a residue of one ton of hard-pressed cake, containing 45 to 50 per cent. of water. The cake so obtained is easily handled, is practically inodorous, air dries very rapidly, and does not again enter into fermentation. It can be kept for any length of time without smell or nuisance. In this condition it can be disposed of in the immediate neighbourhood as manure, for which purpose, as will be noticed, it is available. How far further expenditure, having fulfilled the sanitary requirements of the problem, is justifiable upon a material of so little value can be judged of from the records of past experience; but whatever its ultimate destination, there can be little doubt that the condition in which it leaves the filter



press is that in which it can be most cheaply dealt with, whether for purposes of utilisation or destruction. The woodcut (Fig. 2) shows the arrangement of plant for dealing with the sludge from a population of 30,000, comprising the following apparatus: Air compressor; air accumulator; two sludge filter presses, 3ft. diameter; two sludge forcing vessels, with their fittings and the various distributing pipes for sludge and air; a tip truck and tramway for the removal of the pressed cake discharged from the machines. The cost of such a plant with the requisite boiler power (about 10 h.p. actual) is about £1,000. Thirty tons of wet sludge can be easily pressed into cakes, containing 50 per cent. of moisture, equalling six tons, or one-fifth of the original bulk, consisting of five charges from each machine, of 12 cwt. each, in ten hours. The labour required is about two-thirds of the time of two men. The cost of the operation determined from actual work, extending over two years at Coventry, amounts, with all expenses included, to sixpence per ton of wet sludge, or half-a-crown per ton of pressed cake, and on a larger scale this expense will be somewhat less. The following works may be quoted at which this method is pursued, and where, in some cases, operations have been going on for upwards of two years:—

Croydon Rural Sanitary Authority)	Baldwin Latham,
High Wycombe .....	Esq., C.E.
Coventry .....	The Rivers Puri-
Leyton .....	fication Assoc.
Blackburn .....	General Scott's
	Ammonic Fim-
	us Process.
Aylesbury .....	Native Guano Co.

It may also be added that Messrs. Mansergh and Melliss have recommended its adoption in their lately published report to the Lower Thames Valley Main Drainage Board.

#### SEWAGE SLUDGE AS MANURE.

The idea of converting sewage residues into manure yielding a profit upon the whole operations of sewage treatment, has for some years been rightly viewed as fallacious, and allusion need hardly be made to the failure of the many plans tried with this object. There is, however, no reason why sewage sludge, when deprived of its water, should not be so used, and although too poor to pay the expenses of carriage to a distance, when filter-pressed, it can be profitably employed by the farmers in the immediate neighbourhood. At Coventry this has proved to be the case, for whereas the partially air-dried sludge had either to be given away or got rid of at a charge to the works, the filter-pressed sludge is purchased by the farmers at 3s. 6d. per ton and upwards. It is, however, by no means put forward that this should be the object of so treating the sludge—that must ever remain a sanitary necessity—but as occasion offers it is evident a reduction of the working expenses can by this means be effected, especially if a little care be exercised in handling it after it leaves the filter press. Air-dried it parts with its moisture readily, and the amount of water can thus easily be reduced to 20 per cent., in which state it can, if desired, be easily ground. Being so deficient in those elements which constitute the value of a manure, it cannot of course be taken upon the same basis as artificial manures. There is, however, no doubt that it is fully equal to, and in some cases more valuable, according to analysis, than farmyard manure. Having regard to the fact that the usual price of the latter

never exceeds one-half its analytical value, sewage sludge, with which it fairly compares, should be valued on the same basis. The following examples are selected from results given by the sludge from different systems of treatment, taking into consideration the nitrogenous matter and phosphates only. Nitrogen, calculated to ammonia is taken at 6s. per unit. Phosphoric acid calculated to neutral phosphate of lime at 6d. per unit. The resulting values are shown both for cake containing 50 per cent. moisture as coming from the filter press and also cake air-dried down to 20 per cent. moisture:—

Sludge from Treatment of	Pressed Cake, 50% Water.			Air-dried Cake, 20% Water.		
	Ammonia.	Phosphate of Lime.	Value per Ton.	Ammonia.	Phosphate of Lime.	Value per Ton.
			s. d.			s. d.
Sulphate of Alumina and Lime .....	0·81	1·81	6 0	1·36	2·9	9 7
Lime .....	0·5	2·06	1 0	0·80	3·3	6 5
A B C Process .....	0·69	0·65	1 6	1·10	1·01	7 2
Hansons' Process; Alkali Waste and Lime .....	0·5	0·98	3 6	0·80	1·57	5 7
M. and C. Process; Lime, Carbon, Soda, and Perchloride of Iron .....	0·62	0·86	1 2	1·00	1·37	6 8
Paris Sludge from Liernur's System treated with Sulphate of Alumina .....	2·20	7·5	17 0	3·52	12·06	27 2
Manure made from Pail Emptyings, Goux System .....	0·82	0·96	5 6	1·31	1·57	8 9

From the above analyses it will be seen that the value of the pressed sludge is fully equal to that of farmyard manure; and this statement is borne out practically by the statements of Lieut.-Col. Jones, V.C. "He had tried sewage sludge side by side with farmyard manure, and with 60 per cent. of moisture it was, bulk for bulk, rather superior to the manure."

Before leaving this part of the subject, attention may be directed to the result shown by the manure made from pail emptyings; and it will be seen that even after the expense of separate collection is incurred, the result is of no more value than ordinary sludge, fully establishing from this, as well as from a sanitary point of view, the inefficiency of such processes. The high value of the pressed sludge from the Liernur system of collection is easily understood. Collected by an elaborate pneumatic system, all else save excreta and urine is excluded, and after treatment with a small quantity of sulphate of alumina it forms, when pressed up into cakes by the filter press, a manure far richer than any sludge from water-carried sewage.

#### INFLUENCE OF PRECIPITANTS UPON THE SLUDGE.

A variety of reagents have been used by different inventors for the effectual defecation of sewage, the object in view being the precipitation of the suspended matter and the coagulation of the slimy organic bodies present in solution, as well as in some instances the utilisation of the valuable constituents, five-sixths of which are in solution and pass away in the effluent. Those commonly in use are lime or salts of lime, sulphate of alumina, clay, and salts of iron—and these

are used in a variety of ways, either alone or in conjunction with each other. Phosphates of alumina and lime have also occasionally been used, but their cost, viewed in relation with the inadequacy of the value of the resulting product, of course prohibits their use.

The requirements of a precipitant, as far as regards its effect upon the sludge, are threefold: (1) Rapidity of subsidence of the precipitate formed; (2) the production of a precipitate of minimum bulk with maximum defecation; (3) the sludge obtained should part with its water readily.

In order to determine the most suitable reagent for this purpose consistent with cost, we carried out a series of experiments some time ago, some of the results of which are graphically shown by the curves in Figs. 3 and 4. The substances used were lime, sulphate of alumina, and salts of iron, alone and in different amounts, as well as in conjunction with each other. To determine the effect of these upon subsidence, a standard solution of concentrated sewage was made, containing 20 per cent. of the Paris sludge previously referred to. Measured quantities of this solution were placed in wide tubes all of the same size and depth; the rates of subsidence for different quantities of the reagents were noted and the results expressed by the curves shown in diagram No. 1, Fig. 3. To determine the readiness with which the different precipitates so formed would part with their water during filtration, the contents of the tubes after complete subsidence were agitated and thrown on to filters of the same size and shape. The filtrates were received into graduated measuring tubes, the quantities passing through the filters in a given time being accurately noted, and the results so obtained expressed by the curves shown in diagram No. 2, Fig. 4. The following particulars refer to both tables of curves, the percentages of the reagents being expressed upon the thick sludge originally taken:—

Curve.	Reagent used.	Quantity used.
C <sub>1</sub>	Lime	1%
C <sub>2</sub>	Lime	5
A <sub>1</sub>	Sulphate of Alumina	1
A <sub>2</sub>	Sulphate of Alumina	5
(A and C) <sub>1</sub>	Sulphate of Alumina and Lime	1
(A and C) <sub>2</sub>	Sulphate of Alumina and Lime	5
Fe	Ferrie Chloride	1

Referring to Fig. 3, we learn the maximum amount and rate of subsidence within a given time is that due to the addition of equivalent proportions of sulphate of alumina and lime, and that the precipitate produced by chloride of iron is almost as rapid and the precipitate is less bulky. Referring to Fig. 4, we find that lime-treated sludge parts with its water with no more facility than the untreated sludge. That the sludge from sewage fully precipitated by sulphate of alumina, or equivalent proportions of sulphate of alumina and lime, parts with its water the most readily. An interesting point as to the action of lime is clearly shown in Fig. 4: the coincidence of the curves A<sub>1</sub> and (A and C)<sub>1</sub>, A<sub>2</sub> and (A and C)<sub>2</sub>, points to the conclusion that the lime has little effect upon the filtration, its action being explained by Fig. 3, viz., as a weighty body assisting the rapid subsidence of the sludge.

#### CONCLUSIONS.

The following are the conclusions submitted as a result of the present paper: (1) Every effectual system of sewage disposal is accompanied by accumulations of sludge. (2) That of all the systems employed for the disposal of the sludge, that by means of properly-constructed filter presses offers the most advantages. (3) That pressing the sludge enables it to be disposed of as a weak manure, and that it is at

least equal in value to farmyard manure. (4) That precipitation by sulphate of alumina and milk of lime seems to be the best method of obtaining a rapid subsidence, and a sludge most easily filtered.

#### DISCUSSION.

The CHAIRMAN said he had no doubt they had all listened with great interest to this paper, which put forward most clearly and graphically the most recent progress in this very difficult investigation. Those who had paid attention to the subject would appreciate the truth of the statement that the only thing to be done with sewage was to treat it with some precipitate before putting it on stand, without which the sewage irrigation became an abominable process, and nearly everyone had been met with the almost hopeless difficulty of dealing with the sludge. The paper was also interesting in showing the exceedingly skilful manner in which the special difficulties of filtering a most inconvenient body could be overcome. Of course, every fresh substance needing filtration required somewhat different treatment, and special forms had been devised for special purposes. He should be glad to hear the experience of those who had had to deal with sewage sludge. Whether London would ever set up a sufficient number of presses to dispose of the vast quantity of the metropolitan sewage he did not know. It must be remembered that they had not only to deal with the sewage below London Bridge but that above. This was a matter of vital importance, and could only, in his opinion, be dealt with by the system which had now been advocated, for from all he had been able to hear, he did not think there was any more efficient system of filtration than that now described, and he could speak from considerable practical experience.

Major FLOWER said as the chairman had referred to the pollution of the river Lee at Luton, he was happy to say that the suspended matters in the sewage of Luton were precipitated by lime and clay, and the effluent was afterwards passed through a sufficient area of land; therefore, they might drink the water with perfect impunity. He really thought the proposed mode of getting rid of sewage sludge was most valuable, having seen a great deal of it, for he had spent the last thirteen years of his life entirely on this very filthy subject. The disposal of the sewage sludge was, in fact, the very head and front of the difficulty in treating sewage. At the beginning of the paper Mr. Hutchinson had drawn attention to the different modes of disposing of sewage. It might be somewhat egotistical in him, but he was proud to say he was the first man who advocated the chemical treatment of sewage, and afterwards its utilisation on land. That system was first carried out at Enfield, because the irrigation farm, which was on low-lying land, became such an abominable nuisance. The Enfield people had unfortunately, contrary to his advice, taken to irrigation again, for the company which undertook to treat their sewage by Whittread's dicalcic phosphate process had broken up, and they again found how horribly deficient irrigation alone was, especially when, as at Enfield, the sewage was poured into a water-logged stratum. There the water-table of the country was only 4ft. below the surface. At Tottenham they employed a method which was very fair in its way, but the sludge was a most abominable difficulty, and he believed the very excellent system of filter-pressing sludge was about to be brought into use there. He saw this press in its early days at West Ham, had watched it thoroughly in its progress, and was pleased to find it had arrived at such a point that he thought engineers might really, without stretching their consciences, recommend its adoption on a very large scale.



Mr. DOUGLAS A. ONSLOW had great pleasure in testifying to the efficiency of S. H. Johnson and Company's presses. He had been connected with defecation of sewage by means of precipitation at Coventry for the last seven years. When they first commenced work there they attempted to get rid of the moisture contained in the sludge by means of Milburn's drying apparatus, but the cost of doing so was so extravagant that they were compelled to abandon it, and for want of anything better they ran it into large open tanks, and allowed it to dry as rapidly as it could be evaporated by the air. This was an extremely inefficient way of doing it, and was objectionable in every sense. They made several efforts in a small way with filter presses before meeting with S. H. Johnson and Company, using a somewhat similar form of press, in which, instead of compressed air, they used pumps, but the objections proved very great, and it was found impossible, at any reasonable cost, to get satisfactory results. Fortunately they then came into communication with Messrs. S. H. Johnson and Company, and since their presses had been put up the only difficulty they had had was the breaking of the diaphragms which had been alluded to in the paper, and that was remedied in the way described. This having been accomplished, the pressing had now attained such a degree of efficiency that they had been able to press in the course of one week, working night and day, no less than 476 tons of wet sludge. The necessity for that arose because the presses were insufficient to deal with the total quantity made daily, in an ordinary day's work, which thus caused an accumulation rendering it necessary at times to work night and day. Were it not for that, it would be almost impossible to carry on the operations which, as he contended, had been successfully carried on for the last seven years at Coventry. At any rate, they had been able to deal with the sewage of the whole population of Coventry, 45,000 to 46,000, with the assistance of an area of eight acres of land for the filtration of the effluent water. He was satisfied that the only possible way of dealing with this question was by means of chemical precipitation first, and wherever possible afterwards the filtration of the effluent through land. There were many instances where it was impossible to get land even of sufficient quantity to filter the effluent water from the tanks, and therefore it became necessary to know what was the best possible precipitant to be used for the purpose of getting such an effluent as might be discharged with the least possible harm into the river or ordinary water channels. The paper had dealt most admirably with the details of the presses, and after 2½ years' experience at Coventry they were thoroughly satisfied with them. Mr. Hutchinson had said that they got 3s. 6d. a ton and upwards for the compressed sludge, but as he did not wish anyone to be misled on that point, he ought to say that they got from 2s. to 2s. 6d., but the farmers sent from eight to ten or twelve miles to fetch it. He did not think it was possible to hope ever to realise anything profitable in the shape of returns for the sludge, the manurial value being too small; but still, even if the returns were small, it was something to be able to get rid of that which until lately had been an incubus, which, when it came to many thousands of tons per annum, became a source of great anxiety.

Dr. C. R. A. WRIGHT asked what was the proportion between the amount of nitrogen contained in a given quantity of the original sewage which was carried away by the effluent and the amount retained in the sludge, and the same with regard to the phosphoric acid.

Mr. T. W. B. MUMFORD said the question had

occurred to him that it was all very well to talk about the disposal of sludge and running off a clear effluent afterwards, but he could not understand how large centres were going to be dealt with, where the population was very large, and the district was subject to heavy rainfalls. How would it be possible to get the whole of the rain-water accumulated over a large area dealt with in this way? This appeared to him to be a weak point in the process. There would be at times enormous quantities of water coming down the drains and mixing with the sewage, which would have to go somewhere, and therefore, however perfect the process might be for dealing with a certain amount of sewage, and with a certain area, there would be times when it would be impossible so to deal with it effectually.

Mr. CRESSWELL, in answer to the last speaker, said that most engineers were now agreed that a separate system of sewers was absolutely necessary, no rainfall being allowed to enter them. Major Flower had said that at Enfield they had used chemical precipitation followed by treatment through land. It seemed to him that such a system was unlikely to destroy the noxious qualities of sewage, because nitrifying germs were readily affected by chemicals, more especially antiseptics.

Major FLOWER asked if it did not come to this, that chemical treatment, after all, was simply mechanical, leaving the land to deal with all the putrescible organic matter in the effluent. He knew Colonel Jones's farm very well, and his practice was to leave the sewage for a very long time in his tanks, which were of a special construction, and there get rid by deposition of the matters in suspension, just in the same way as, on a larger scale, it had to be done by chemical means. It appeared to him that chemical treatment, after all, was only mechanical in its operation.

Mr. CRESSWELL said that Colonel Jones, V.C., told him emphatically that it was advantageous to treat sewage by subsidence simply, without addition of chemicals, and then to run the effluent on to suitable land, in which state it could be readily attacked by the germs.

The CHAIRMAN, in proposing a vote of thanks to Mr. Hutchinson, said it must be borne in mind that at best sewage precipitation was only a palliative, the land itself must take what remained in solution; but those who had seen the working of a sewage farm would know the ineffable abomination of applying unfiltered sewage to land in large quantities. Sewage precipitation never could carry down all the phosphoric acid, still less all the nitrogen; but it rendered it possible for the land to deal with the effluent afterwards, if land could be had.

Mr. CRESSWELL wished to explain that the question was as to the chemical action of land. It did not act simply as a mechanical filter, but as an agent for the oxidation into nitrates of the nitrogen of sewage; and the point he wished to emphasise was that the liquor must not be put on the land in such a state as to preclude the action of the germs.

#### REPLY.

Mr. HUTCHINSON, in reply, said, with reference to what the chairman had said of the overwhelming quantity of sludge which would be accumulated from the sewage of the metropolitan area, he thought, in the course of a few years, steps would be taken to carry out what he had stated as desirable in the paper, viz., the removal of the solid matter, admitting only the purified effluent into the river. The engineering difficulties could easily be got over, and were not by any means overwhelming. His firm would not have the slightest hesitation in entering into a contract to press up the whole of the sewage sludge of London every 24 hours, and he should feel

the utmost confidence in their power of being able to so deal with it. With reference to Major Flower's observation respecting the sludge nuisance at Tottenham, he thought probably if he went there in a few months' time he would not have the same complaints to make, as he hoped before then the sludge would be reduced to the inodorous condition as the specimens he had shown. Mr. Onslow had made one remark which he should not like to go forth without some little qualification. He said the reason they had to work their machinery night and day was that the presses were not able to deal with the whole of the sludge accumulated daily. Now he wished it to be distinctly understood that this did not arise from any fault of the presses themselves, but on account of the inadequacy of the size of the plant compared with the quantity of sludge they had to deal with. That plant was put up for dealing with about 28 tons a day, and, as Mr. Onslow had pointed out, they had actually pressed up 480 tons in one week, which was equal to 80 tons a day, working 24 hours, or 40 tons working 12 hours, clearly showing that the presses did 33 per cent. more work than they were originally intended to do. With reference to the error which he had pointed out as to the price obtained for the pressed sludge, he would say that perhaps they did not get what they should for it. In such cases much depended not on the actual value of the material, but on the idea you could give people of its value, and probably, if a little pains were taken, after the sludge came from the presses, to give purchasers an idea of its value, they might be able to get a few shillings more per ton for it. He suggested that a little care should be taken. It should not be thrown into a heap, and the farmers be told that they could have as much of it as they liked at 2s. per ton; but it should be removed in pieces from the presses, carefully handled, and suitably stacked under cover for air-drying. Such a course would considerably add to its value. The manager of the Coventry works had recently told him they had just entered into a contract for 1,000 tons at 3s. 6d. per ton. With reference to Mr. Munford's observations, the process now brought forward did not pretend to enter into every phase of the difficulties connected with dealing with sewage, but simply the difficulty of dealing with the sludge. No matter what the rainfall was, whether it were 1in. or 40in., there would be a certain quantity of sludge to deal with, and the reply Mr. Cresswell had given to that question was perfectly correct. The conditions of modern sewage disposal decidedly pointed to the direction of keeping the whole of the solid matter and excreta entirely separate from the surface drainage and rainfall, because in dealing with a less bulky and dilute material it rendered the process of purification and disposal much simpler and less costly. In all those towns which were not yet fully sewered a separate system of surface drainage would be carried out for taking off the whole of the rainfall, which would pass into the watercourses sufficiently pure without any treatment. In those towns which already had a complete system of sewerage the plan was being adopted of providing storm overflows, the sewers being so constructed that when a certain quantity of water had accumulated in them, the remaining part due to the storm, which would be comparatively clean water, flowed over without any treatment into some stream. Mr. Cresswell said that Colonel Jones informed him that it was quite possible to deposit the whole of the solid matters in sewage by subsidence alone, but he would simply refer Mr. Cresswell to the observation of a philosopher in olden times, who demonstrated the possibility of moving the world.

he only had a lever long enough and a fulcrum on which to place it. The case of the simple subsidence of the whole of the solid matters in sewage without chemical treatment was somewhat analogous. Given sufficient tank area, and an unlimited length of time for the sewage to pass through those tanks, it would probably be possible for the whole of the solid matter to be separated by subsidence. In Fig. 3 the curve of subsidence of untreated sewage was shown to be practically a straight line, and it was coincident with the datum line. It was known by experience to sanitary engineers that it was practically impossible to settle out the whole of the putrescent matter in the sewage without chemical treatment. In reply to Dr. Wright, he believed about five-sixths of the nitrogenous matter passed away in the effluent, and that only one-sixth was precipitated in the form of sludge. The nitrogenous matter so precipitated was of the most objectionable character—namely, that present in the shape of albumenoids, which would most readily enter into putrefaction. That which went away in the effluent was principally ammonia salts, ammonia, and nitrates. The amount of phosphoric acid precipitated depended altogether upon the treatment pursued, the chief difficulty being the very dilute form in which it was present in the sewage. It was, however, quite manifest that such a treatment as that of sulphate of alumina and lime would throw down the greater part of the soluble phosphates as the insoluble phosphates of lime and alumina, and such a result was given in actual practice.

## THE PORTER-CLARK PROCESS.

BY J. H. PORTER.

THERE are few industries independent of the character of water supply, whether as regards the production of steam or the excellence of the results of various operations, technical and artistic. In both the presence of the carbonates of lime, magnesia, and iron in the waters employed is a continual source of loss, often very great, as in the incrustation of boilers, which is inadequately appreciated. Dr. Clark's water-softening process has necessarily but limited application, in consequence of the large space required on the one hand for mixing and setting tanks, and on the other, the time required for the subsidence of the precipitated carbonates. The desirability of applying the process, chemically perfect as it was, suggested the idea of filtration for the removal of the suspended carbonates. Many patents were taken out for devices for effecting this object, and so far the most successful one has been that known by the title of this paper. In 1877 Sir Frederick Bramwell read a paper before the British Association on the subject, and Mr. James Duncan was the first to practically test the process on a large manufacturing scale at his sugar refinery. Other refiners have followed suit, as also some railway companies, as the London and North-Western Railway at Camden, Willesden, and Liverpool; paper makers, dyers, brewers, and others. At first open tanks for continuous softening and filter presses for continuous filtration were used, as at the Silvertown Indiarubber Works. For greater economy of space, and essentially to avoid the necessary second pumping, was devised the method of working under pressure, by which, indeed, pumping is altogether avoided when working under gravitation.

Figs. 1 and 2 represent the plant in use at the locomotive sheds of the London and North-Western Railway at Camden, for treating 7,000 gallons per hour continuously, and where water of 17° to 18°



hardness is reduced to 4°. The water treated at Willesden and Camden is pumped from deep wells in the chalk near Watford, and reaches those stations by gravitation, and with a pressure of 60lb. to the inch. Except when treating very small quantities of water, continuous agitation is maintained within the vessel of the apparatus. This pressure is utilised in working a Ramsbottom water engine or motor, which Mr. Webb (head of the locomotive department at Crewe) suggested for the purpose. The exhaust water from the motor is conducted (still under

agitation, and passes out and upwards to the tallest vessel, in which saturation of water with lime takes place. The particles are prevented from settling out by the slowly-revolving agitator. The saturated and almost clear water passes from the upper part of this vessel (which has a sampling cock at due elevation) to the dome on the shorter vertical vessel, by a pipe, fitted with a regulating valve. Hard water from the main is also entered here, working the motor on its way. The two streams thus mix in this dome at about equal pressures, and decomposition is effected

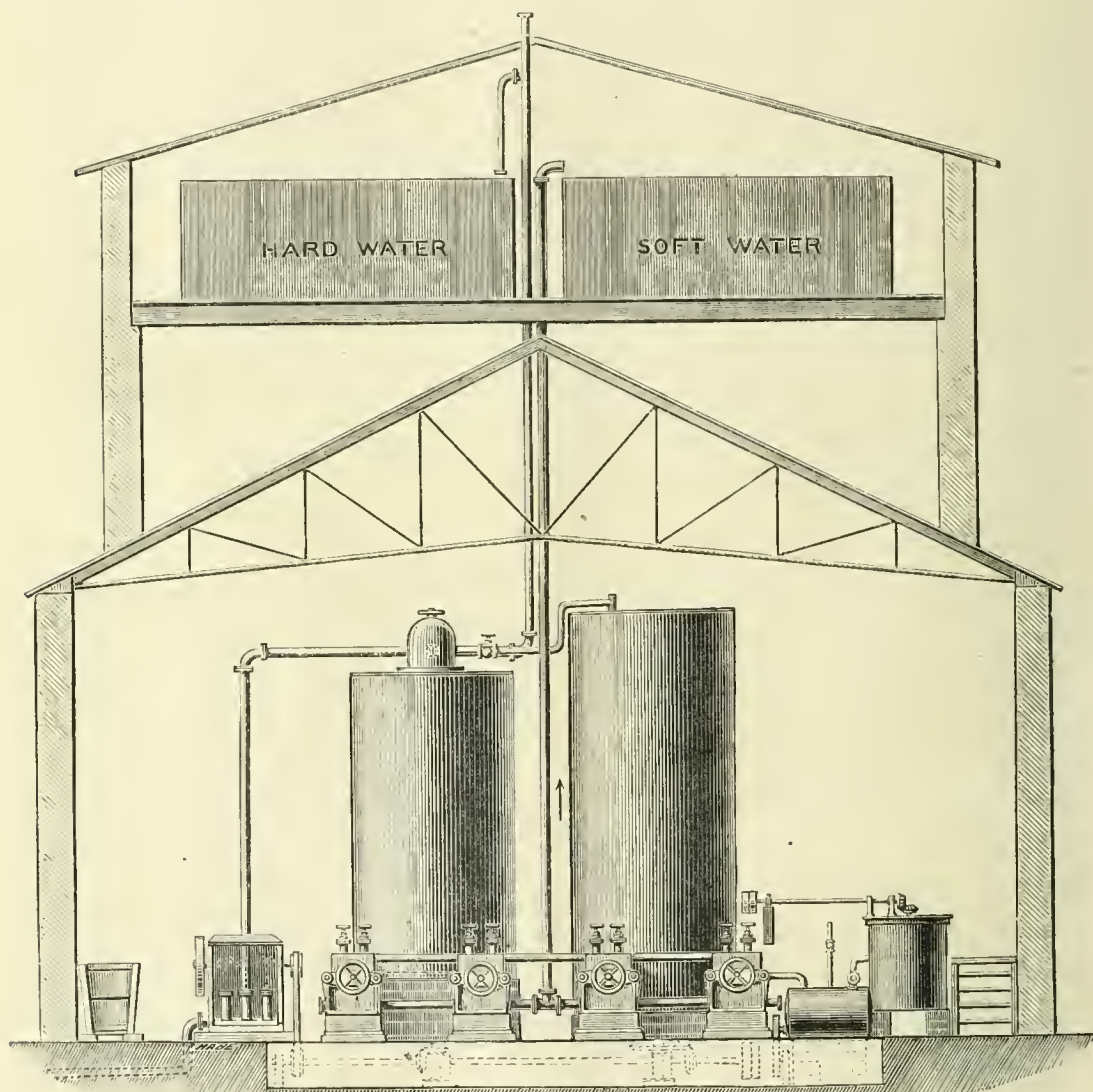


FIG. 1.

pressure) into and through the system of vessels and filters. Leaving the separated and suspended carbonate of lime in the latter, it passes up to the elevated service tank. The diagram exhibits two large vessels 7ft. in diameter, and respectively 13ft. and 17ft. in height. In addition is a horizontal cylinder or churn, 2½ft. diameter by 4ft. long. All three vessels are fitted with agitators, driven by shafting worked by the Ramsbottom motor, but so geared as to give to the agitator in the tallest vessel a speed of about ⅓th of the other two. In the churn, slacked lime is kept

completely and rapidly. The suspension of the separated particles of carbonates is continued through the shorter vessel to the connected filter presses, in which they are arrested. (A frame containing separated carbonates of "great whiteness from the Camden plant was exhibited.)

In the case of certain waters containing much organic or aluminous matter, the older direct method of Dr. Clark was useless. The improved process is successfully applied to water drawn directly from the Thames. In such cases it is necessary to employ pure water

(as from the companies) or previously treated water for the production of lime water. This consideration has not affected the economical use of the improved process. (A frame containing carbonates and suspended matters of a dark umber colour was exhibited.) It is scarcely necessary to add that any intelligent workman soon becomes *au fait* with the tests indicating the due proportions of the hard and lime waters during the softening process. Nor is it needful to indicate the possible adaptations of the process in a meeting of this Society. But one at any rate may be referred to, viz., the prevention of "incrustation" in boilers, a subject which has been ably treated in the Section by Mr. Ivison Macadam. To large steam users this has an estimable value in the prevention of frequent stoppages for chipping out, for occasional repairs, and considerably in the saving of fuel. The engineer of the Manchester Steam Users' Association, in his report to the committee, points out the desirability of throwing out the impurities of feed waters before entering the boilers, and not in them. This gentleman expresses the hope that the Porter-Clark process might be made available for the removal of sulphates in some similar

disruption from the presence of confined gases. With regard, however, to brewing, it has been found that water deprived of its carbonate of lime more readily acquires "brewer's" hardness by solution of "sulphate" where artificial hardening has to be resorted to. The immense saving in numerous directions which would result from the use of an efficient system of softening water on a large scale needs no demonstration. We venture to think that we have gone some way to the fulfilment of this end by a process which is calculated to accomplish this effectually and economically; for in general terms it may be said in this as in many other cases the larger the scale of operation the smaller the cost. In specific terms one may say the space occupied is small, and the clarification immediate. Suffice it to say that in the following towns using the process the figures indicate the reduction in hardness:—

Canterbury .....	26.3 to 4.9
Caterham .....	21.2 to 1.1
Tring .....	26.3 to 3.2

## DISCUSSION.

Mr. B. E. R. NEWLANDS said this process had been worked at Mr. Duncan's sugar refinery, Clyde Wharf

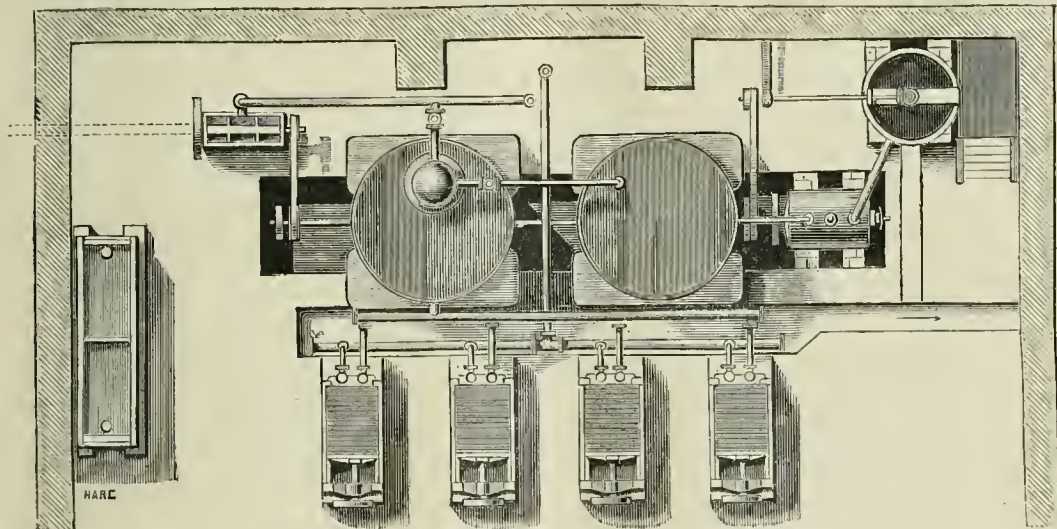


FIG. 2.

manner. With regard to this latter point there are considerations of cost, which are rapidly lessening in consequence of the cheaper production of certain apparently necessary chemical agents.

In one of the London works in which the process is now used, and 400 tons of fuel are disposed of weekly, 60 tons less were formerly burned immediately after periodical chipping out. Mr. Macadam, in his paper, states the loss of fuel as 30 per cent. Mr. Rowan gave 15 to 60 per cent. as the range of loss. At a paper mill, by an apparatus similar to that of the London and North-Western Railway Company, a saving was effected of 14 cwt. out of a consumption of 7½ tons daily under the old conditions. It may not be uninteresting to mention that occasionally the delicacy of the water-mark of the Bank of England note-paper was impaired by the presence of small granules of carbonate of lime, which became mechanically retained in the delicate wirework designs. This trouble is now obviated by the adoption of the Porter-Clark process. One need scarcely refer to the use of certain waters in breweries, but for the production of ice it has been found that the greater the freedom from carbonic acid the water employed can be the more transparent the ice and the less liable to

Victoria Docks, for several years. In fact, the first practical apparatus was erected there, and all that Mr. Porter said with regard to its successful operation had been fully borne out by actual results. With suitable water, such as that supplied by the East London Water Company, which was good compared to that obtained from the artesian well at Clyde Wharf, the hardness could be reduced to an extremely low limit. With Clyde Wharf water they were not quite so fortunate, because it contained a large quantity of alumina, magnesia, and organic matter, and these impurities prevented the removal of the hardness to some extent. Still there was doubtless a great improvement in the water after treatment. He saw on the table an old friend of his, namely, a piece of lead pipe which he gave to Mr. Porter a year or two ago, taken from a pipe leading to a condenser. This pipe was almost closed by incrustation. The cause of this was not quite certain, as a rapid current of water continually ran through it; but it may have been due to the precipitation of calcic carbonate, owing to the removal of carbonic dioxide by the vacuum. Every three or four months the pipe used to stop up, but since the Porter-Clark process had been employed no trouble had been experienced.



Great credit was due to Mr. Porter for the admirable way in which he had carried out his recent improvements. At Clyde Wharf they had his earliest form of automatic arrangement for regulating the amount of lime water, which answered very well, but the new apparatus, such as had been described, was no doubt a great advance in the right direction.

Mr. J. J. EASTICK said he could bear testimony to the benefit of the action of the Porter-Clark process upon water taken from the Thames between Blackwall and Woolwich. Unfortunately the composition of the Thames water fluctuated considerably, so that we had a slight difficulty with regard to the proportion of lime water to be added, but that was the fault of the water, not of the process. If the proportion of lime was too little there was great difficulty in filtering, but by so arranging the lime valve as to always have a slight excess this difficulty was removed, and no great objection was found to the presence of this lime in the boilers. Another advantage was in the use of the water while warm, which was found to render the filtration much more effectual. In making the lime water at Silvertown they used the water which had been treated, or else they used the East London Company's water; but they had used the Thames water direct, and found it to answer very well.

Mr. JOHNSON was also pleased to bear testimony to the efficiency of the working of Mr. Porter's apparatus, which he had seen on various occasions at different works. In one he had looked into, a tank of water was softened by this process, and he noticed that besides removing the carbonate of lime it rendered the water so clear that he could conceive that it would be possible almost to read a newspaper at the bottom, a depth of 24 ft.!! The water lost that peculiar yellow colour due to the organic matter of the ordinary water supplied by water companies, and had the blue of pure water; and he thought that ought to go a long way to recommend the adoption of this process by water companies. In the sixth report of the Commissioners on the pollution of rivers it was reported by Dr. Frankland that the treatment of water by Clark's process ought to be adopted, and that the companies ought to be compelled to use it before they were allowed to raise additional capital. Mr. Porter had not alluded to the various recommendations of the Commissioners in favour of soft water, such as the advantage in the making of tea, coffee, and cocoa. He believed that from one-half to two-thirds of the tea used in London could be saved by this means. There would also be an immense saving of soap in laundries, for it had been shown that a very small quantity of lime (one farthing's worth) was equal to a saving of about 30s. worth of soap. All these things went to show what great economy there would be by the use of soft water for household purposes. These matters might seem trivial, but when taken on a large scale they became important. He should like to know what would be the cost of this process on the waterworks scale. Mr. Porter had stated that it would cost about 1d. per 1,000 gallons on the scale of a quarter of a million gallons a day, which would come rather expensive on the scale of 10 or 12 million gallons. Dr. Frankland stated in his report that it could be softened for about £1 per million gallons, and he should like to hear if any calculations had been made on that point. It was also stated in the same report that the carbonate of lime precipitated had a certain commercial value, which might be taken as a set off. It was put down at 15s. per ton, and it was said that about three tons of carbonate of lime was produced for every ton of lime used in the process. If this were so it would more than pay the cost of the lime, but no doubt this would greatly

depend on the quality of water treated. Where the water came from deep wells in the chalk very little organic matter would be separated, and then the material ought to have a market value greater, he should think, than that of ordinary whitening. He had examined under the microscope the lime precipitated by this process, and comparing it with ordinary whitening he found that it was perfectly crystalline, whereas the latter was amorphous. Possibly this might have some effect on its commercial value. He would also ask Mr. Porter if he had adopted the silver test for testing the water for excess of lime after it had been subjected to this process, which he thought was more practicable for a workman than by boiling the water or using Dr. Clark's soap test.

Dr. MESSEL said: I have received some favourable reports from the Continent of a process in which magnesia is employed instead of lime as in Clark's process. By its means the lime, combined with sulphuric acid, is precipitated as well as the carbonate. Sulphate of magnesia is formed at the same time, and it has no injurious effect on the boiler. If burnt dolomite could be employed the process ought to be as cheap as it is said to be effective, and Mr. Porter's apparatus seems well adapted to try the experiment. Perhaps he has already done so, and I should be glad to hear his experience or opinion of the process.

Mr. W. LANT CARPENTER said that a few years ago he had some experience in the working of the process of purifying water from sulphates, which was brought under his notice in Germany. It consisted in the simultaneous use of either milk of lime or lime water and chloride of barium, and in agitating the water with these. Practically it was a process especially adapted to water to be used in boilers, and the most convenient mode of agitating the water was found to be the use of a steam injector carrying in a blast of air with it, which gave very effectual agitation, and also warmed the water. By bringing the water to about 105°F. the precipitate settled very rapidly. No filtration was necessary for this purpose, and a boiler fed with it worked for a year continuously, without showing the least sign of incrustation. It might be urged that chloride of barium was an expensive salt to employ, but its cost was saved many times over in the diminished consumption of coal necessary to evaporate the water in a clean boiler, with no non-conducting material interposed between the source of heat and the water.

The CHAIRMAN said this was a most interesting description of a process which certainly ought to be much more widely known. Clark's process was inapplicable, except in special circumstances. It might do very well for the vast storage reservoirs of a water company, but nowhere else. Mr. Porter had shown an exceedingly neat application of the same chemical principle to the ordinary needs of manufacturers or smaller places.

#### REPLY.

Mr. PORTER said: With regard to the cost of treating very much larger quantities of water than those required for manufacturing purposes, he had not yet dealt with a larger quantity than one ton of water per minute. No doubt if they had much larger quantities to deal with some economy could be effected in the relative capacity of the vessels, in the cost of the filter presses, and also in the amount of labour employed, and probably the cost would come down to half the present cost. What he always told the water companies was that they charged the public for filtered water but did not filter it. The whole point between the public and the water companies was that the water was not sufficiently filtered.

Another point was this: the water companies grumbled about paying a farthing or a halfpenny for a thousand gallons, but he contended the water he used cost him about 3s. per 1,000 gallons; and taking houses of a higher rent than his, such as those in Belgrave Square, the figure would probably work out very much higher still. With regard to the apparatus lending itself to the use of other reagents, there was no doubt it would do so, and this matter had been lately brought to his attention by one of the insurance companies. Nearly all sulphates of lime and magnesia could be treated with chloride of barium, but the great difficulty of these things was the cost of material. Lime only cost about 15s. a ton.

Mr. MEADE said he understood magnesia stone could be got for 3s. 6d. a cwt.

Mr. PORTER said that seemed a reasonable price, but whether the magnesia was in a form suitable for the purpose he was not chemist enough to say, but it would be objectionable if the magnesia, like soda, remained in solution, as it might cause "priming" in the boiler. He had never tested the commercial value of the precipitated carbonate. From the London and North-Western works in Liverpool they took it to Crewe to mix with clay for brickmaking. At the India-rubber works it was used instead of whitening for paying over the cables.

## Liverpool Section.

Chairman: E. K. Muspratt.

Vice-Chairman: Prof. J. Campbell Brown, D.Sc.

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Hudson A. Binney.

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W. Douglas Herman.

E. Milner.

E. W. Parnell.

C. Symes.

H. Tate, jun.

G. T. J. Wells.

Hon. Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be made to Edward George Ballard, Queen's Park, St. Helens.

The next Meeting of this Section will be held on Wednesday, March 5th, 1884, at Seven o'clock, when the following will be the order of proceedings. (1) Paper by Dr. Eschellmann, on "The Loss of Nitre in the Manufacture of Vitriol;" (2) Paper by Mr. E. W. Parnell, on "The Action of Nitrates on Alkaline Sulphides."

UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL,  
January 7th, 1884.

MR. E. K. MUSPRATT IN THE CHAIR.

## NOTE ON THE PRINTING OF NATURAL INDIGO ON CALICO.

BY ROBERT BOURCART.

THIS style of printing, as it is done at the works of Messrs. Schlieper and Baum, Elberfeld, has been very obligingly communicated to me by Mr. Adolf Schlieper, who ordered a great variety of pieces to be printed in my presence, and has given me the following indications, with permission to communicate them to those interested in calico printing. Printing by natural indigo has long been practised, and many works use a process described in the treatise of Persoz, which consists in throwing down white indigo with tin crystals, and printing in an acid state. The combination of white indigo with tin is decomposed when the pieces are suspended in the air, and indigo fixes itself on the fibres. Nevertheless, by this process only light blues can be obtained—sky blues; but dark blues, as produced by dyeing in the indigo vat, cannot be

made by this process. Artificial indigo made with orthonitrophenylpropionic acid, which is called in the trade propionic acid, is far more expensive. It can only be used for small designs, and even if its price were not a hindrance a very dark blue could not easily be obtained. Moreover, when it is printed in somewhat heavy designs, the unpleasant smell of xanthate of soda, with which it is fixed on cloth, reappears again, notwithstanding the most energetic washing with hot water or sodium carbonate. The process of Messrs. Schlieper and Baum allows of printing a blue colour with natural indigo. This colour can be made light or dark as is wanted. It is identical with dyed blue, called dip blue, for shade, and costs less for the same darkness. It has, therefore, a great future before it. The details which follow are all necessary to make a good colour. They must be followed exactly, because the smallest fault makes the printing fail; but if the work is carefully done a very regular and fine blue is obtained. First of all, a special arrangement is wanted. Erect a small shop to print the blue, with one or two machines: surround it with partitions to avoid draughts; heat it in the winter, like an apartment. Each printing machine must be accompanied by a steam chest, and by a small drying machine to dry the cloth before printing. It must be, besides, furnished with a special drying apparatus, with hot air blown from a roof's blower. The doors of the workshop must slide up and down, to avoid all displacement of air. (See Fig. 1.)

The principle of the Schlieper and Baum process is as follows: Glucose in presence of alkalis is an energetic reducing agent chiefly in the heat, and in contact with steam at 100°C. The reducing agent acting upon finely ground indigo transforms it into white indigo, which is dissolved by the alkali which is present, and is immediately fixed on the vegetable fibres, which are possessed of a special affinity for reduced indigo. Water is then powerless to carry off the indigo. Cotton has been dyed in the same manner as hanks of wool or silk, which have been dipped into a solution of aniline colours. By washing the glucose and soda are then carried off, and soon, in contact with air, the blue develops itself. It is oxidised again, and then withstands perfectly all soapings, alkalies, and light—the latter, however, bleaches slowly indigo. For printing indigo a very thick colour is made, containing very concentrated caustic soda, indigo in the state of a paste—that is, very finely ground with water—and a thickening (mixture of roasted Indian corn starch and Indian corn starch). This colour must be very thick, because it must be simply deposited on the surface of the fabric, and not permeate it. The cloth is previously padded in a solution of glucose, then carefully dyed, so that the alkaline colour and glucose are superposed, but not mixed in the printing.

To dry the pieces the greatest care must be taken. They must be dried slowly, as much as possible with hot air, and not be allowed to remain in contact with hot plates or cylinders, otherwise the soda will destroy the indigo before reduction takes place. It must also always be verified if the dried piece contains a trace of moisture. If it is completely dry, or if it has been too much heated for a single moment, the colour is much weakened. The temperature of drying must never be above 75°C. Steaming is carried out immediately after printing. For that purpose a special box is used, which is placed immediately behind the printing machine, and care is taken that the pieces be dipped in an atmosphere of pure steam completely deprived of air. The soda then attracts water, penetrates the cloth prepared with glucose, and reduces the



indigo, which the fibre absorbs immediately. The pieces must come out of the steaming a brown colour with an olive tint. If they are yellow it is because they have been dried too much before steaming, and they will only give in that case a very pale blue. After steaming, the pieces are immediately washed with special care, much water being employed, the soda and glucose being carried off. There still remains on the cloth a trace of soda, which causes the blue to oxidise quickly in the air, and therefore is beneficial. The cloth is again washed with water,

# I.—INDIGO ALONE.

## Preparation of the Printing Colour (Standard Colour).—Receipt—

25 kilos. indigo.  
100 kilos. water.  
50 litres caustic soda, 70° Tw.  
58.53 kilos. solid caustic soda, 90 per cent., sufficient quantity to change the 100 kilos. of water into alkali, specific gravity 1.35; together with the 3.75 litres water which are employed to prepare the printing colour.

Grind in an indigo mill 25 kilos. indigo (standardised

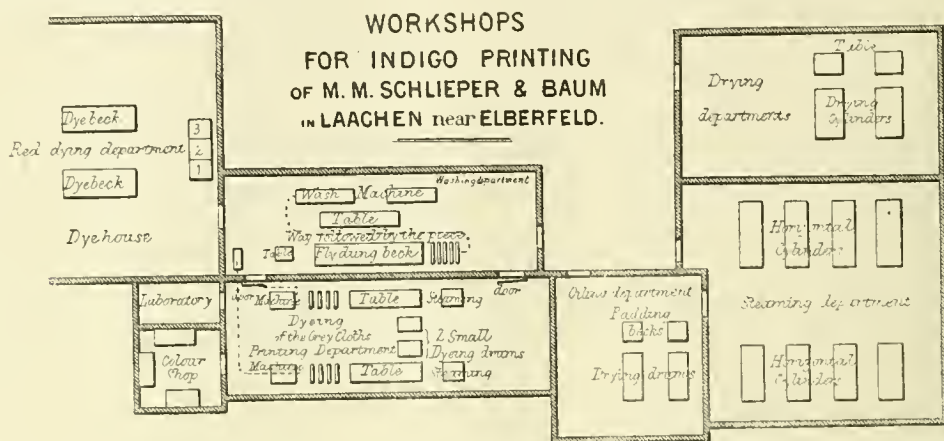


Fig. 1.

The Root blowing machine is in the washing-room. The dotted lines, which in the designs come from the apparatus, show the way followed by the hot air at the top of the printing-room. A is the only piece marked, which is on the ceiling; it represents five rotatory winces of polygonal shape.

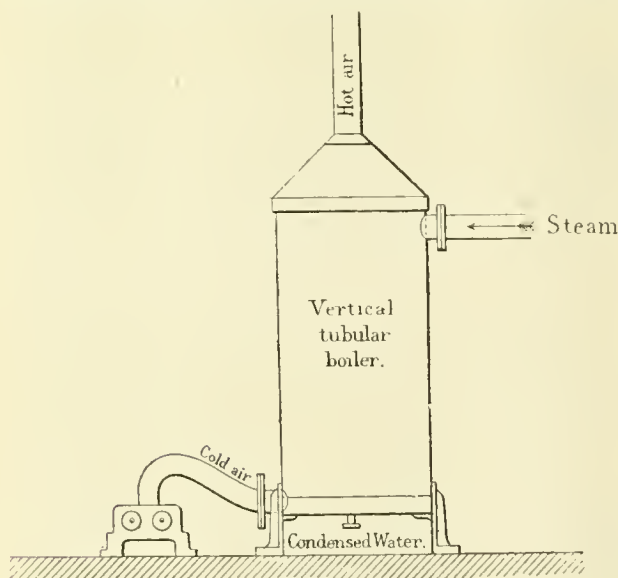


Fig. 2.

and afterwards dried. During the drying the oxidation of the blue is completed. To avivate or brighten this, it is treated at the boil with a weak solution of caustic soda, which replaces soap with advantage. (See Figs. 2 and 3.)

The following is a description, with details, of the steam indigo process as it is practised by Messrs. Schlieper and Baum, at Elberfeld: (1) Indigo alone. (2) Indigo on turkey-red. (3) Indigo on turkey-red mordant. (4) Resists under steam indigo, two-colour designs, and half resists.

at 70 per cent. of pure material by mixtures of various kinds together) with 50 to 70 parts of water. The best qualities are those which are tender. Java is the best of all, and costs eight shillings per pound. When the indigo is ground, which occupies two days, enough water (which is first used to rinse the mill) is added to make up 100 kilos. Then the 125 kilos. are put on a balance having on one side weights and on the other an iron pan suspended in the midst of an empty trough. This trough can be, when needed, filled with cold or tepid water. Then

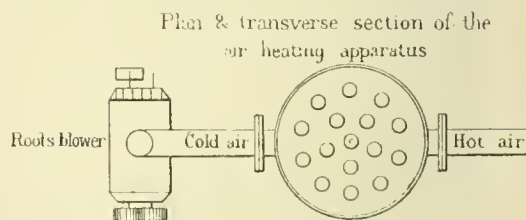


Fig. 3.

the alkali is added (sp. gr. 70° Tw.), and then, at once, 58·53 kilos. solid caustic soda, broken up in lumps varying in size from that of the fist to that of a nut. After that it is stirred with a spatula of iron, and care taken that the temperature does not ascend higher than 40° C. If the mixture becomes hot, cold water is introduced into the trough, and finally the latter is filled with tepid water at 40° C. The standard colour, when ready, is put into cast-iron boxes of about 25

Or per kilo. of colour—

	Grms.
British gum.....	55
Indian corn starch.....	27

*Remark.*—The colour must be such that the caustic soda which it contains, compared with the total quantity of the colour, stands at 65° to 70° Tw. These two limits must not be passed, neither must they be fallen short of. Caustic soda, 70° Tw., of specific gravity 1·350, represents 440grms. solid commercial

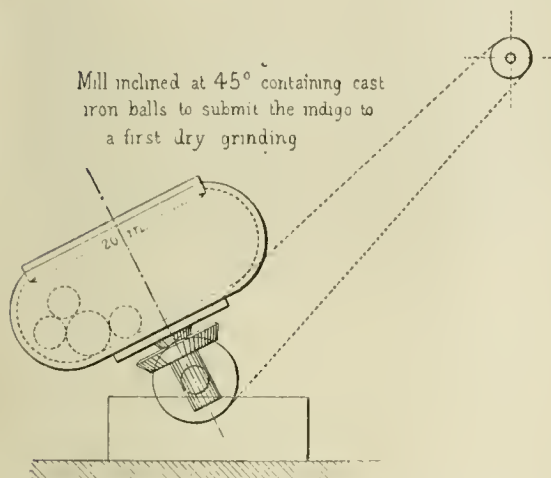


Fig. 4.

kilos. capacity, of which the lid lutes itself in a circular gutter containing water, intended to prevent the access of air, which contains carbonic acid, and which combining with soda would give crystals. The printing colour is also kept in these boxes. Messrs. Schlieper and Baum make 250 kilos. standard colour at one time. The vessel in which this mixture is made is therefore very large. It is hooked on to an

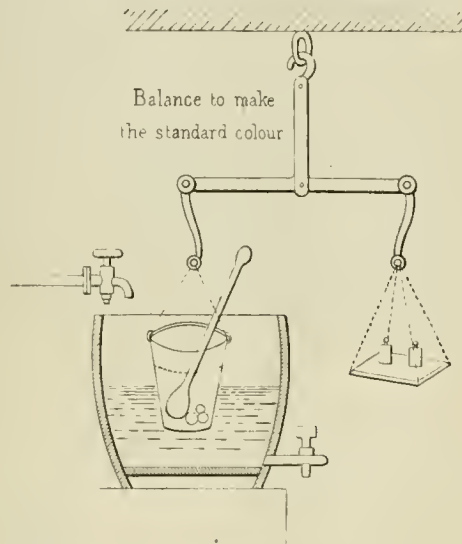


Fig. 6

caustic soda to the litre. To make the printing colour, 3 kilos. pale British gum (roasted starch half soluble in water) are mixed with 1·5 kilo. Indian corn starch and 3·750 kilos. cold water, in a pan standing in a cold water-bath, then half a litre caustic soda, 70° Tw., are added, and stirred for ten minutes with a wooden spatula; then again half a litre is added, and the stirring is repeated precisely as before, though

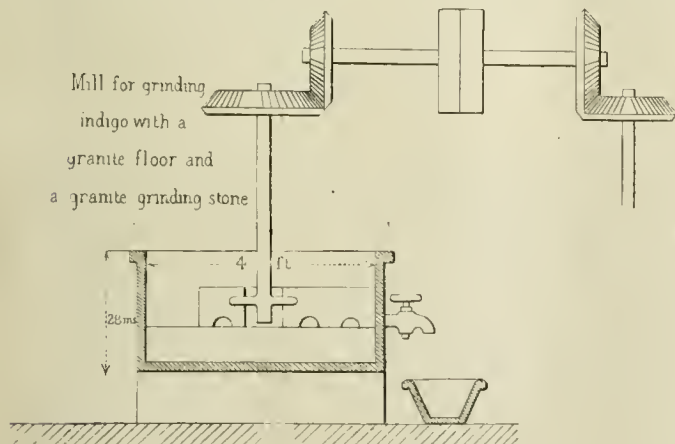


Fig. 5.

iron balance, and is suspended inside a wooden trough made of cask sides. (See Figs. 4, 5, 6, and 7.)

#### Printing Colour.—

	Grms.
Pale British gum.....	3,000
Indian corn starch.....	1,500
Water.....	3,750
Caustic soda, 70° Tw.....	19,000
Standard colour.....	27,000
Total.....	54,000

Vessel with hydraulic lid

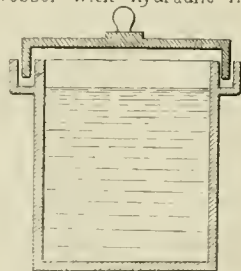


Fig. 7

very slowly. As it is useless to stir it all the time, a man can mix three colours at once. Finally the standard is added. The quantity of soda for a very dark colour will be, for instance, 19 kilos., and that for the standard 27 kilos. These proportions can be varied according to the intensity of the blue wanted, but the sum of the quantities of caustic soda (70° Tw.) and standard must always be 46 kilos. The colour with 18 kilos. of standard is still



very dark, and costs 2s. 3½d. per piece of 66 yards. The thickening per piece is reckoned at 4d. The colour with 33 kilos. of standard is nearly black. Colours are named by giving them a number showing how many kilos. of standard are contained in 54 kilos. of printing colour. When all the soda as well as the standard is added to the thickening, which takes about two hours, it is heated quickly to 70° C. in the water-bath and well stirred to make the mixture more homogeneous; it is then rapidly cooled in the cold water-bath. Afterwards the colour is placed in the above-described iron pots. Before printing it is strained through sieves, by hydraulic pressure, or through bags, between two wooden sticks. The colour must always be very thick, which does not prevent it printing well, because it is also very smooth. When heated a little it becomes more fluid and oily. This result is also obtained by stirring with a spatula. This colour keeps for several months, but after such lapse of time it must be heated very slowly, and stirred strongly before using it. The colour must always be printed slightly tepid, and rollers and boxes must be made lukewarm before using them.

from the roller, and touches directly the warm plate. The back cloth and gray cloth are dried behind the machine by passing between hot plates; but the piece, on arriving at the top of the room, undergoes a special kind of drying. In the other room is machinery blowing hot air, which sends air at 75° C. through a tube of the size of a common stove pipe. At the top of the room, under the ceiling, is a threefold box, somewhat wider than the piece, and nine yards long. The cloth goes three times this length, descending each time from one box into the other, the warm air accompanying it; then it goes out, and is deposited in folds on a table. There the workman must continually try with his hand what is the amount of moisture still present, and quicken or slacken the speed of his piece to suit the quickness of the drying. For blue alone, it is dried in such a manner that to the touch it is very slightly moist and gummy; for blue on turkey-red, on the contrary, it must be dried more, and to the touch must be nearly dry. Same recommendations for blue with sulphur resist, so that the resist cannot run into the blue.

*Steaming.*—The cloth must be cold before steaming,

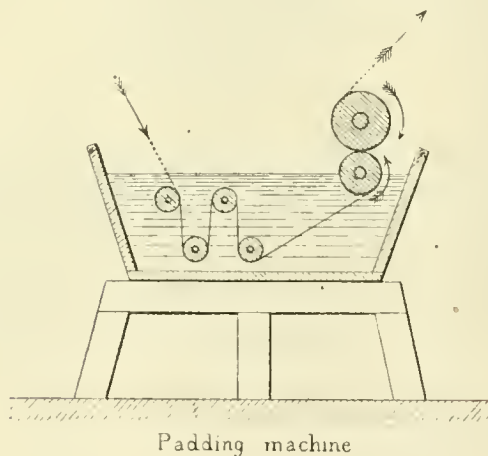


FIG. 8.

*Preparing of the Cloth in Glucose.*—(Solution of glucose, 12° Tw.)—The cloth must be passed into a solution of glucose by means of the preparing machine. The solution of glucose (12° Tw.) contains 250grms. to the litre of water. Care must be taken that the cloth goes several times up and down the rollers, before being squeezed out. For cloths dyed in red, special attention must be given to them becoming thoroughly saturated. Therefore a hot solution of glucose is used, because these kinds of cloths, being oiled, are very difficult to wet thoroughly. After that they must be dried and wound up. In preparing, attention must be given that lint does not remain sticking on the cloth, because it would be very difficult to remove afterwards with the revolving brush. The cloth must be dried a second time before printing, glucose being hygroscopic, and the piece, when printed, must be very dry, and rather warm. (See Figs. 8 and 9.)

*Printing of the Colour.*—The printing colour is very thick, and the printer must stir it constantly with his spatula. The printed cloth moves along a lukewarm steam plate, which ascends to the ceiling. It leaves its back cloth a few inches

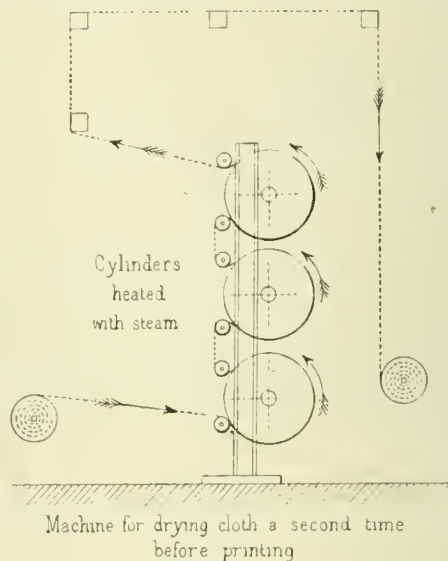


FIG. 9.

because if it is warm the soda does not condense enough water in passing through the chest. It must be steamed immediately after printing. The steam chest is of wood. It is 5ft. high, without counting the base, which is of iron, and contains water, above which are two or three sieves. Above the steam chest is a steam plate, which is 10in. wider on each side. The steam, after having passed in this plate, descends into the lower part of the chest and comes into the water. On its way it receives, on both sides, steam from two small pipes placed at the entrance and at the exit of the pieces. These pipes are placed in such a manner that the piece immediately before entering, and after coming out, passes over them. They protect it from the moisture of the steam, which is condensed by the air in coming out of the box. The steam plate must also, for the same purpose, reach 10in. out over the entrance and the exit of the cloth. The piece, before and after steaming, ascends a steep incline, not to be moistened by the steam, which, coming out of the sides, ascends in a kind of funnel of canvas into a chimney, which draws it outside. The steam which condenses on the canvas cannot fall upon the pieces, there being other sheets of wet canvas below the

former, and two gutters for carrying off the dropping water. In the steam chest are rollers below and above. The piece ascends and descends in twenty seconds, and then passes out and falls in folds into a canister. All the machinery is in a special room, screened with glass partitions all round, to avoid draughts, and the doors slide up and down. There are in this room two printing machines, producing 25,000 pieces of 66

minutes. During this time they pass five strong waterjets which throw water from above. There are five large compartments furnished with rollers at the top and at the bottom. Between the boxes there are squeezing rollers, which forward the pieces from the one into the other. The dung beck is 8 yards long and 4ft. high. Nine-tenths of alkali are here washed off, and what remains

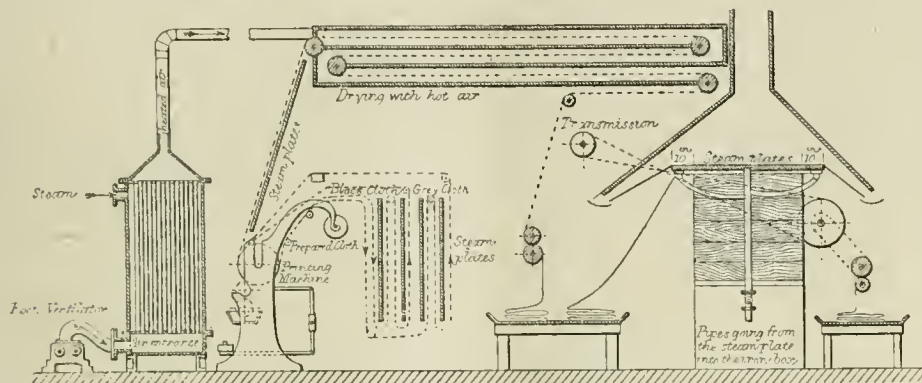
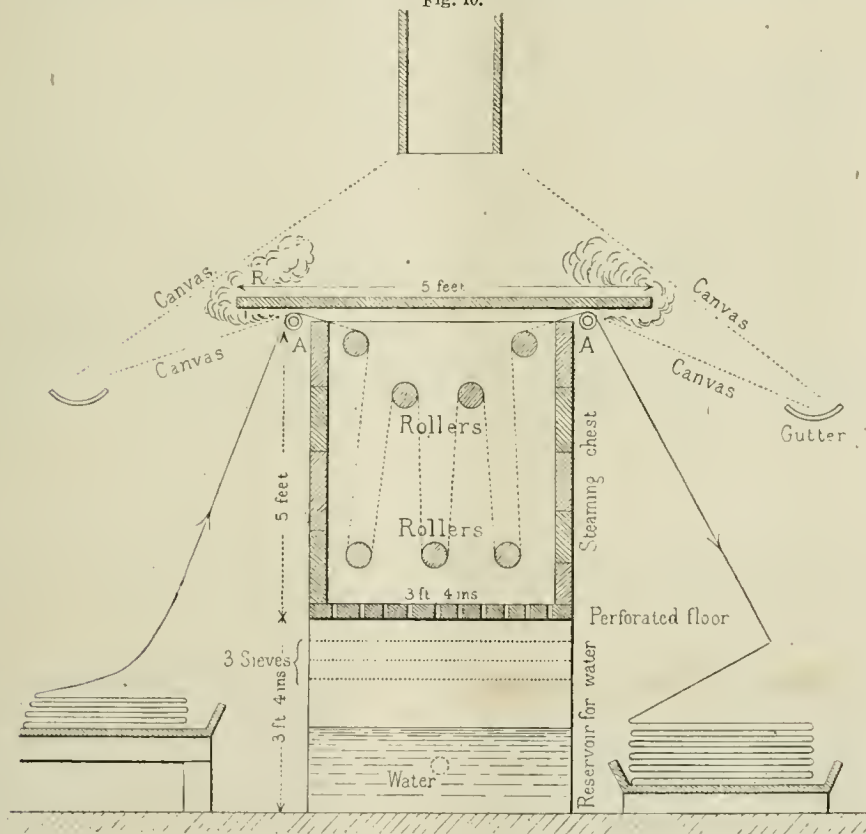


Fig. 10.



Apparatus for steaming the indigo

Fig. 11.

yards in the year, chiefly plain pads for discharge work with pigments, albumen, and chromate of potash. (See Figs. 10 and 11.)

**Washing.**—When the pieces are steamed they are carried into the washing-room on the other side of the glass partition. There they pass first in the open state through a large fly dunging machine (see plan of works), in passing through which they take two

can do no harm, because the piece is at once forwarded to the washing machine. There would be more alkali taken off if the dung beck contained rotatory winces, which would beat and shake the cloth, just as they do in the endless open soaping machine. At the end of the dung beck the pieces are pressed between the two last squeezing rollers, so that the water will not prevent



the oxidising action of the air. They then pass over five cylinders with rods fixed at the ceiling, whose work it is to shake them in the air, after which the pieces fall in folds on the ground. When about 35 yards have been passed, the pieces are put in communication with a washing machine of the usual construction. The pieces are previously formed into a rope, by passing through several porcelain rings, and then washed successively through two washing machines of middle size, for five pieces of 66 yards. Then the cloth is dried on a drum, where the oxidation of the colour is completed. It is not yet lively, and to aviate it the cloth is passed into a boiling solution made of 500 litres of water and 5 litres of caustic soda, 45° Tw. The colour then becomes bright, because the soda carries off a brown colouring matter. Besides this, soda carries off less blue than soap does, and is cheaper. The cloth is again washed and dried, and the pieces are ready.

red. But for this, acid treatments are required, which all kinds of turkey-reds could not withstand. To print the blue, the red pieces are prepared like the white ones, with a very hot solution of glucose, and by passing repeatedly on rollers before squeezing out (see sketch of the preparing machine), so that a complete moistening can be attained. After printing, the pieces must be dried more than for blue on white cloth, because all the strength of the alkali is required to destroy the red, but a very slight trace of moisture must be felt still. Then they are washed in the fly dung beak and washing machine and dried on hot cylinders, the same as before. They afterwards pass through another dung beak (see plan of works), which is in the dyehouse. This has three compartments. The first contains sulphuric acid (10½° Tw.) The cloth passes, in seven or fourteen seconds, to the shade of blue required. The first shade has a slight purple cast; the second is bluer but a little

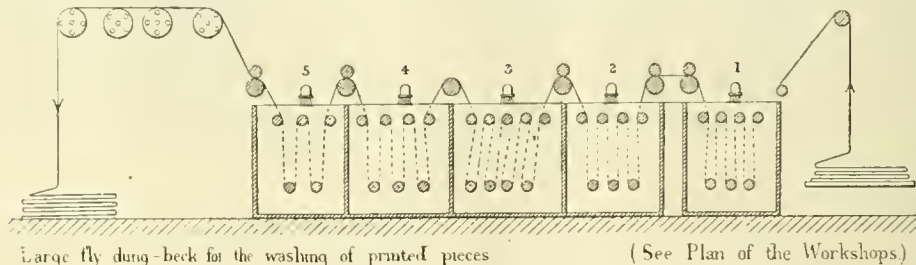


Fig. 12.

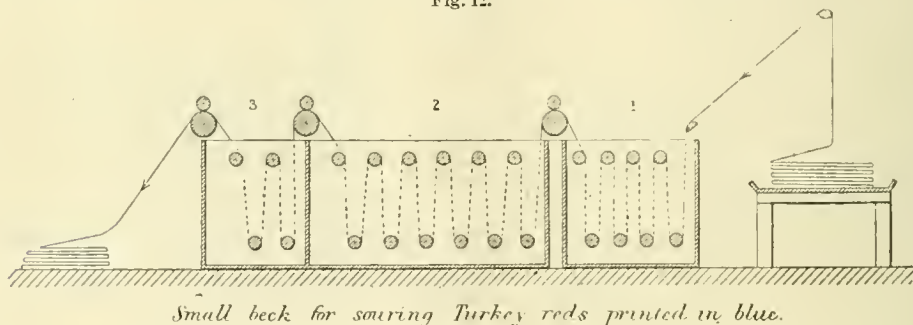


Fig. 13.

Blue 27 is the ordinary colour, and costs about 3s. 1½d. per piece of 66 yards, whilst the dip blue of the same intensity costs 3s. 7½d. This blue can be used for discharge styles; but if plain pads are wanted, a colour of half strength should be printed twice over, as this would make the shade more even;

Sample No. 1.



Printed indigo, with reserve in the engraving or the roller. but all operations, preparing of cloth included, would be needed to be repeated twice over. (See Fig. 12 and sample No. 1.)

## II.—INDIGO ON TURKEY-RED.

This process depends upon the fact that by suitable operations the alkali of the blue destroys turkey-

red. lighter, whereas the Turkey-red ground withstands perfectly.

The second compartment is somewhat larger, and contains water, to wash off the acid. The third is smaller, and contains carbonate of soda 3° Tw. Its alkalinity is scarcely felt with the

Sample No. 2.



Turkey-red cloth, printed in indigo

hand. This last compartment is very small. It has two pairs of rollers, the middle one six and six and the first one three and three. Between them are squeezing rollers, which forward the pieces from one into the other. The goods are afterwards strongly soaped at the boil with soap, when the latter becomes quite purple, and finally the pieces are washed and dried. (See Fig. 13 and sample No. 2.)

## III.—INDIGO ON TURKEY-RED MORDANT.

This process aims at sparing alizarin, because where there is blue the mordant is destroyed before fixing any colouring matter. (a) Preparing of the mordant. (b) Printing of the blue. (c) Discharging of the mordant. (d) Dyeing in red. (e) Brightening of the red.

(a) *Preparing of the Mordant.*—The mordant is an aluminate of lime. First, aluminate of soda is printed on, which is made as follows: Take dry alumina from the "Chemische Fabrik zu Niemburg an der Wesel." It is a nearly pure hydrate of alumina,  $(Al_2(OH)_6)$ . Forty kilos. of this substance are heated for three hours with 64 litres of caustic soda at  $35^\circ$  Bé (about  $50^\circ$  to  $55^\circ$  Tw.), and then diluted to the volume

that it is dunged in a chalk beck for twenty minutes, to give back to the mordant the lime which sulphuric acid has taken away.

(d) *Dyeing in Turkey-Red.*—For dyeing, very large troughs are employed—about  $5\frac{1}{2}$  yds. long, 5 ft. high, and 7 ft. wide, without compartments. The pieces pass open, same as in the fly dung beck.  $1\frac{1}{2}$  grm. of alizarin (10 per cent.) are added to every litre of water, and for every grm. of alizarin (10 per cent.) there are added, first, 4 grms. saturated lime water (1 to 800), and afterwards again as much in the course of the dyeing. The liquor has a brown colour, and as the pieces pass along, alizarin and lime water are added, according to the quantity, known beforehand, which each piece absorbs in passing through the dye beck. The

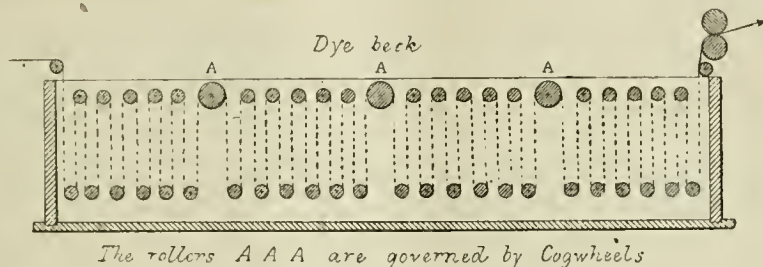


Fig. 11.

of 300 litres with water. This soda must correspond litre for litre with hydrochloric acid (sp. gr. 1.15). After this, 8 litres of hydrochloric acid are added. In this manner there are still 56 litres caustic soda not neutralised in the liquor. It is then diluted to the volume of 620 litres with water. This aluminate, to be good, must be still able to receive 30 c.c. of hydrochloric acid, (1.15 sp. gr.) without losing its clearness; if not it must be exactly adjusted by adding either hydrochloric acid or caustic soda. Then for every 4 litres of mordant 1 or, better, 2 litres of water are added. It is blotched and dried on drums. The cloth turns yellow. It is passed for twenty minutes through the ageing-room system of Walter Crum—that is, in a moist and warm atmosphere, where pieces ascend and descend on rollers. After that they are left lying in bundles from 12 to 36 hours, and then washed in a

duration of the passage is from 3 to  $3\frac{1}{2}$  minutes, and the pieces come out dyed at the other end. For dark pads the cloth is dyed twice over, but they are avivated between the two dyeing operations. The temperature of the liquor is  $90^\circ$  to  $95^\circ$  C. Before being brightened the red is of a dark-brown colour, but it is very able to combine with the acid soap which we describe further on. Care must be taken not to bring the liquor to a boil, because in that case the red would not combine any more with the brightening soap. This is a very interesting operation, because it offers all the advantages of rapid work, fastness, fine shade, and cheapness united. (See Figs. 14 and 15.)

(e) *Brightening.*—The cloth is padded in acid soap. The latter is used instead of oleine. It is made by preparing, first, the soap of castor oil and soda, and neutralising afterwards exactly half of the soda

Steaming cylinder with its wagon

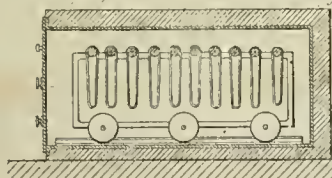


Fig. 16.

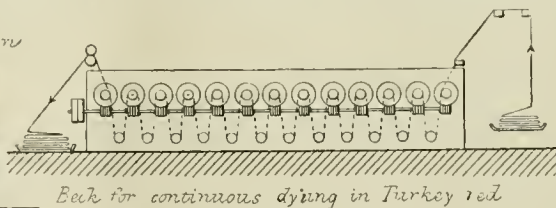


Fig. 15.

fly dung beck and pass through a washing machine for half an hour in a lukewarm bath containing a good quantity of chalk. They are afterwards washed slightly with water, much chalk being left on the cloth. After that the pieces are heaped together for 24 hours in the wet state, then the chalk-bath is repeated, and they are allowed to lie for 24 hours again. The mordant is then completely transformed into aluminate of lime. Lastly the pieces are well washed, and they are ready for dyeing. If chalk were not employed, the red or the mordant would not at all be able to withstand the action of sulphuric acid.

(b) *Printing of the Blue; and (c) Discharging of the Mordant.*—The blue is printed in the same manner as on a dyed red, and it undergoes the same operation of washing and passing into soures. After

with hydrochloric acid. Acid soap is a liquid which floats on the surface of the trough where it is made, and below is the solution of sodium chloride, which has been formed by adding hydrochloric acid to the soap ley. The red cloth saturated with this liquid is dried on drums, and then steamed. Steaming is continued for one hour, at  $\frac{1}{2}$  lb. pressure, in horizontal cylinders arranged in rows, and into each of them is introduced a small wagon, running upon rails, and furnished with ropes, made of goats hair, on the top. On these ropes the pieces rolled on themselves, are suspended, so that they hang off about  $3\frac{1}{2}$  ft. After steaming, they are again soaped and washed. The steaming, for an unknown reason, is always somewhat hurtful to the blue; therefore a very dark colour must be employed, that it may be still dark after having lost somewhat



of its strength. For instance, blue No. 27 will give a blue No. 20 or 18, which is still of good depth. (See Fig. 16.)

#### IV.—RESISTS UNDER BLUE.

*White Resist.*—This is done with precipitated sulphur, which is bought from De Haën in Hanover, and which is prepared by means of the polysulphide of sodium. This precipitated sulphur, which contains always sand, is ground in a small mill with a solution of British gum. It is afterwards thickened with British gum by taking 140grms. sulphur per litre. This sulphur, when indigo is printed on and steamed, is changed into polysulphide of sodium, which prevents the fixation of the colouring matter. To receive this resist the pieces must be well dried before printing on the blue, that the design be neat. (See sample No. 3.)

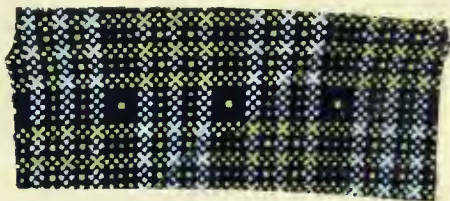
Sample No. 3.



Cloth printed in indigo and sulphur resist, and covered in indigo.

*Yellow Resist.*—200 grms of cadmium chloride are mixed with the above colour. In contact with sodium hydrate, sulphur dissolves and forms yellow cadmium sulphide. (See sample No. 4.)

Sample No. 4.



White and yellow resist under printed indigo.

*Resist with a Mordant for dyed Alizarin Red.*—This is more difficult to make, because only acid or neutral drugs can be added to sulphur. If the required mordant is employed, which would be aluminate of soda, the rollers and copper-faced doctors would be soon destroyed, copper sulphide being formed. With acid mordant, on the contrary, alumina formed from the acetate would be at once washed off in contact with the caustic soda dissolved from the piece in the first beck of the fly dunging machine, at the commencement of the washing. A special arrangement must therefore be devised. In the first compartment of the dung beck (see Fig. 12, No. 1 compartment), a weak solution of arseniate of soda and sal-ammoniac, to the extent of 200 grms. per litre, are added, and care is taken to add enough to compensate for the quantity which the piece carries away during the course of the washing, notwithstanding the rollers which squeeze the liquor out. One litre is lost for every eleven yards of cloth. In this operation a double exchange takes place immediately between the soda of the cloth and the sal-ammoniac. Sodium chloride is formed and ammonia is set at liberty, and this ammonia does not dissolve alumina. When the liquor smells too strongly, ammonia is neutralised with hydrochloric acid. The piece passes afterwards through water (compartments 2, 3, 4, and 5) as in other resists. Generally, how-

ever, there is in No. 5 a weak solution of silicate of soda to give the cloth an alkaline reaction, and so allow the blue to oxidise quickly in the air. When there is not much blue printed on, and consequently little alkali, there is no need of using any sal-ammoniac. Compartments 1, 2, 3, and 4 contain, then, only running water, and No. 5 a solution of silicate of soda (3° Tw.), which acts as a dung for alumina, and prevents it from running. The red is dyed as described before. The mordant must be somewhat concentrated, and contain about 10 grms. per litre of tin crystals. (See sample No. 5.)

Sample No. 5.



Printed in white and red mordant resist, printed in indigo, dyed in red, oleined, steamed, and soaped.

*Designs with Two Rollers (Red and Blue).*—This style resembles very much the one above. The best plan is to use alkali red mordant (aluminate of soda), because acid mordant marks off on the second roller for blue, and precipitates itself on it as hydrate of alumina, fills the engraving, and consequently makes a light blue mark on the piece, repeating the design on the first roller. Alkali red mordant, on the contrary, answers the purpose, if care be taken not to steam more than 20 seconds; then the pieces are passed through sal-ammoniac or silicate of soda, according as there is more or less blue on the cloth. These styles of red and blue are very expensive when it is needed to employ sal-ammoniac in the beck.

*Half Resist.*—If colour 0 be employed (i.e., without any standard colour in it), and this be steamed, all the glucose is destroyed, and is no more able to fix any blue. But if, on the contrary, an alkali of weaker strength is printed on, the glucose will only be partly destroyed. On that rests the printing of all shades of half resists. After steaming, the pieces must simply be dried on the small cylinders for drying prepared pieces; then the blue is printed as on other resists, and then steamed and washed. (See sample No. 6.)

Sample No. 6.



Cloth mordanted for red, prepared in glucose, printed on with strong alkali; steamed, dried, printed in indigo; steamed again, washed, dried, sonred, dunged, dyed in red, oleined, steamed, and soaped.

#### GENERAL REMARKS.

1. The great future of printed indigo depends upon the fact that it is less expensive than dip blue. All discharges which are made on the latter can also be made on steam indigo blue, and in that case it needs not to be printed twice over, as with plain pads.

2. To destroy the blue on stained pieces and bleach them, Messrs. Schlieper and Baum boil them in a small kier with red prussiate and permanganate of



potassium; but it would be easier to bleach them by enclosing them in one of the large kiers in the bleach-house, and heating them there for seven hours, at 50 lb. pressure, with carbonate of soda and resin. In that case they come out at once thoroughly white. This process is also good for pieces dyed in dip blue. It seems that indigo can withstand pretty well strong alkalis, even if warm, at the ordinary pressure, but with three or four atmospheres even carbonated alkalis destroy it.

3. Indigo can be very well printed on linen, and in fact is so printed in England at the present time.

#### DISCUSSION ON MR. BOURCART'S PAPER.

Dr. HURTER supposed the great advantage of the process described was the facility with which the pattern was printed, whereas formerly it had to be dyed. Dr. Bourcart had mentioned the necessity of not having the goods too dry, as otherwise the caustic would destroy the indigo, but what about the cotton itself? He would like to know if the process described would compete with the printing with artificial indigo, or would be likely to supersede it? From the samples shown by Dr. Bourcart it would appear that the very finest patterns could be printed, so that in this respect the new process could compete with the propiolic acid process.

Dr. BOURCART: Formerly there were no means of obtaining a dark shade of indigo, and this was provided now by Messrs. Schlieper and Baum. He had submitted a sample of cotton printed with indigo to the microscope, the caustic soda used in the process having been extremely strong, 65° to 70° Tw. Nevertheless the cloth was not destroyed at all. He had observed, moreover, that the threads were perfectly dyed. Propiolic acid is only used by calico-printers on a very small scale, and then there was a great disadvantage owing to the bad smell imparted to the cloth, which often prevented customers from buying it.

Mr. W. D. HERMAN: Does it retain the smell long?

Dr. BOURCART: Several years, I think.

February 6, 1884.

MR. E. K. MUSPRATT, IN THE CHAIR.

#### THE LANCASHIRE SODA TEST.

The CHAIRMAN announced that Mr. A. Norman Tate, who had promised to read a "Note upon the Lancashire Soda Test," was unable, from indisposition, to attend. In his absence he (the Chairman) would read a letter he had received from Mr. Tate. The following letter was accordingly read by the Chairman:—

"I am sorry that, being confined to the house in consequence of indisposition, I shall not be able to be present to read the 'Note on the Lancashire Soda Test,' and not being able to get access to some papers and journals that I wished to allude to, I cannot send you a full note that might be read by some person else. I would, however, say that my object in reading the note was to call attention to some remarks made by Mr. John Pattinson, analyst, of Newcastle, at the meetings of the London Section and of the Newcastle Section (both meetings are reported in the last *Journal of Chemical Industry*). Mr. Pattinson states that the Liverpool soda test is from 1 to 3 per cent. higher than the Newcastle test. Mr. Davidson, of Newcastle, also stated that he had found differences, and that samples divided into two portions had given different results by Newcastle test and by Liverpool test, Liverpool being 48 per cent. against 45 and 46 in Newcastle. In vol. 32 of *Chemical News*, page 267, Mr. Pattinson made a similar statement, and a long discussion then ensued, in which I took part, when Mr. Pattinson then stated that he did not refer to any tests made by me. Whether this is the case now or not I cannot tell from Mr. Pattinson's remarks, as reported in the *Journal*; but as he speaks generally of 'Liverpool tests,' I wish to say, as one making a fair number of such tests, that the basis on which I work is precisely the same as that given

by Mr. Pattinson, and referred to fully in tables he has published, viz., that the ratio of the commercial test to that based upon the equivalent of soda last adopted is  $\frac{37}{31} = 31.307$  (or  $31.11:31$ ). Mr. Pattinson, in his tables, gives the same ratio, or rather practically the same,  $31.11$  to  $31$ . It is, therefore, difficult to see where, if the samples are really the same, so wide a difference as even 1 per cent. can occur. If such differences are the rule in the tests of one and the same parcel, I would suggest that sampling and not testing is at fault. However, I regret that I cannot be present to discuss the matter fully this evening, but trust I may be allowed an opportunity at the next or some future meeting. I would, however, lose no time in denying, so far as I am personally acquainted with the matter, that these wide differences can exist when careful sampling and testing have taken place, and this care is taken fully, as far as my experience goes, in Liverpool. The practice of reporting in whole number referred to by Mr. Pattinson could not at any time show a greater difference than 0.5, or not fully that, per cent., and this difference is as often in favour of buyer as seller. Further, a chemist must have unbounded confidence in his own skill if he can state positively to 0.1 per cent. the strength of a parcel of many casks of soda-ash or drums of caustic soda that he has sampled and tested. The practice of reporting in whole numbers, as a matter of chemistry, in connection with the commerce of such articles as carbonated soda-ash, caustic soda-ash, caustic soda, etc., is, to my mind, the most prudent, common-sense, practical method. Personally I do not have two standard acids in my laboratory for testing alkalis. I invariably use a strictly normal standard (for too many standards lead to confusion), and when making a commercial soda test I use a special weight strictly in accordance with the figures I have already referred to, and which are the same as Mr. Pattinson's. This also is done to avoid error from using wrong weights."

The Chairman proceeded to say he thought it very important that the Section should express itself upon the remarks which had been made by Mr. Pattinson at meetings of two other Sections of the Society. He was sorry to find that Mr. Pattinson, Dr. Lunge, and other Newcastle chemists, had made the statement that the Lancashire soda test differed from the Newcastle test by some extravagant figure of 3 per cent. He thought that was unjust and that they had not spoken with the accuracy befitting scientific men. One of the advantages of a society of this kind was that they did inculcate accuracy about statements they made. As Mr. Tate said in his letter, the whole difference between the Newcastle test and the Liverpool test could only amount at the outside to one-half per cent., and the methods of testing were the same; standard acid used was practically the same, and therefore any greater difference that might arise must be either from the sampling or from the personal error of the operator. Now he had yet to learn that it was absolutely impossible for an operator in Newcastle to make a mistake. It was probable that a Newcastle chemist, when he came to test a sample of soda ash, was as liable to error as a Liverpool chemist. They were assured by Mr. Tate—and he thought they would receive similar assurances from other analysts in Liverpool—that the method employed was practically the same, that the only difference was in the way of reporting, and it was well that the statement made by Newcastle chemists should be denied authoritatively from this Section. He would be glad to hear any observations that members might have to make.

Mr. HUSON said he had come that night for the purpose of stating that it had been the practice of his firm (Huson Brothers, Liverpool) to base their tests on the assumption that pure carbonate of soda contained 59½ per cent. of soda, which was the custom generally in Lancashire; and on reading through Dr. Hurter's paper some short time ago he wrote to him pointing this out. They decided the letter should not be published, because the remarks were simply incidental, his paper being not on the Lancashire soda test but another subject, and therefore the letter was not sent to the Society's journal. But he believed Dr. Hurter had now brought the letter with him and would read it. He quite agreed with the chairman's remarks about the possibility of Newcastle gentlemen being in fault, and did not see why Liverpool should be altogether to blame for the difference.

Dr. HURTER said shortly after the December num-



ber of the journal was published he received the following letter from Mr. Huson:—

"In the account of the discussion on the subject of international standard methods of sampling and testing commercial products which took place on the 7th ult., which appears in the number of the *Journal of the Society of Chemical Industry* just to hand, and which we have read with considerable interest, we find a statement to the effect that the Lancashire test is based on the assumption that pure anhydrous carbonate of soda contains 59.81 to 59.60 Na<sub>2</sub>O. As a very large proportion of Lancashire-made alkali has for many years been tested by us, we think it right to state that in our case the above figures are erroneous. Our test is, and always has been, based on the assumption that pure anhydrous carbonate of soda contains 59.26 per cent. Na<sub>2</sub>O. This statement, to the writer's own knowledge, applies to a period of over twenty years back from the present."

He had in his paper made the statement that the atomic weight of sodium varied from the year 1810 to the year 1883 in such a manner as to include differences of 59.8 per cent. to 58.4. What he meant to say was that the mere variation of this atomic weight produced quite as great differences as could ever be observable between the Lancashire test and the Newcastle test. He quite agreed with the chairman that the various gentlemen who from time to time had written to the *Chemical News* had been unduly severe on Lancashire manufacturers as well as the Liverpool analysts. He thought Professor Lunge had been very indiscreet in mentioning names, especially in the German edition of his book on the alkali manufacture. It was utterly impossible that there should be 3 per cent. of difference, even if the Liverpool analysts had used the larger equivalents, which they say they had not used. Even in that case the possible difference between the two tests could only be 0.4 per cent., and such a difference as 3 per cent. was perfectly absurd, and pointed to an error either of sampling or manipulation. All he hoped was that the movement now in progress to bring about uniform methods in sampling and testing would end these everlasting discussions about the Liverpool and Newcastle tests.

Dr. CAMPBELL BROWN said he had been an on-looker in this dispute for the last 20 years without taking part in it, but he had felt very strongly the propriety, to use no stronger word, of the proposal which had been made by Dr. Hurter this year, that all persons interested—manufacturers and analysts—should agree to use the proper scientific figure for the atomic weight of sodium, and conduct all tests and estimates of value upon the best and most accurate figures which had been ascertained up to the present time. He hoped the Society of Chemical Industry would make an effort to have a national if not international agreement as to the conduct of tests upon certain methods which were known to be the most accurate.

The CHAIRMAN thought the discussion on Mr. Tate's note would set the matter right, and he thought it was also a very strong argument in favour of what they desired to bring about, viz., some agreement as to the mode of testing, and what methods should be adopted.

#### DISCUSSION ON MR. WATSON SMITH'S PAPER.

The Section then proceeded to the discussion of Mr. Watson Smith's paper upon "The Tars from Coke Ovens, Blast Furnaces, and from the Sutherland Gas Producers," read December 5th, 1883, and January 7th, 1884.

Mr. DOUGLAS HERMAN thought the sample of tar tested by Mr. Watson Smith could scarcely be considered as fairly representing the quality of tar capable of being produced by the Sutherland process, for the reason that the condensation was inefficient, owing to shortness of water. The temperature of the water running from the condenser averaged

143° F., and the liquor in the well 117°. Under these circumstances it could scarcely be expected that benzol, even if formed at all, and the more volatile oils should be present, especially if the large volume of gas, something like 150,000 cubic feet to the ton of coal passing off at this high temperature, be borne in mind. To show the effect of even a trifling improvement in the condensation, he might mention that for some time the production of tar was barely 4 gallons to the ton of fuel; but by slightly increasing the quantity of water to the condenser, and causing some to flow through the hydraulic main, the yield was increased to 8 gallons, and even then there was undoubtedly a large quantity of tar passing away with the gas.

Dr. HURTER spoke of the difference of opinion as to the conditions under which certain tar contained the valuable constituents, benzol, etc., and under which it did not. In some books it was stated that the lower the temperature of distillation the more benzol was got, whereas in other books the opposite was asserted. His own opinion (the reasons for which he gave) was that the view that the lower the temperature the more benzol was produced, was absolutely wrong.

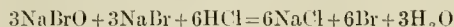
Dr. Hamburger and Mr. Allen (Sheffield) followed

#### REPLY.

Mr. WATSON SMITH: In reply to Mr. Douglas Herman I should not like to say much until I have tested a better condensed sample of tar from the Sutherland producers, but I think I may speak to the strong probability of the lower boiling naphthas in well-condensed Sutherland producer tar, consisting chiefly of lower paraffins and olefines, with a certain small proportion possibly of the benzene group of hydrocarbons. The similar nature of this tar to that of the tar of the blast furnaces, as regards its higher boiling constituents, points unmistakably in this direction. Referring to the remarks of Dr. Hurter, with regard to the possibility of drawing anything like a sharp line, and defining rules for so carbonising fuels as to obtain fixed products, that lies only within narrow limits. Not only must the carbonising temperature giving certain results be kept constant, but the carbonaceous material used must not vary, must also be constant, and even then the products will be to some extent mixed. I will try to illustrate my meaning. The same coal carbonised in closed gas retorts at high temperatures, and giving normal coal tar, would at comparatively very low temperatures give a tar of low specific gravity, resembling the tar of the Scotch shale distillers. The first tar is rich in benzene and its homologues: the latter contains little of these, and olefines and paraffins take their place. Anthracene has disappeared, and in its place solid paraffins appear. Again, if Wigan cannel be used solely for gas-making, whatever temperatures are employed, the tar will be found to contain some solid paraffins deteriorating the anthracene. Hence tar distillers often stipulate in their contracts with gas companies that not more than a certain percentage of cannel shall be mixed with the coal carbonised in the retorts. As regards benzene and its homologues, those high temperatures in carbonising coal in closed spaces, which aid in generating benzene and its homologues; and phenol, cresol, naphthalene, and anthracene, if they are carried higher and become very high temperatures, then benzene, toluene and xylene become scarcer, phenol shares their fate, whilst naphthalene and anthracene increase and become abundant. But though in distinction to the highest temperatures, those found to yield benzene and its homologues, with carbolic acid, most abundantly, may be termed "lower temperatures" yet, in

comparison with the lowest temperatures of destructive distillation at which paraffins and olefines are almost exclusively yielded, they are justly termed "high temperatures." In reply to Dr. Hamburger, it is true that certain tar constituents cannot be so exactly measured and estimated in a laboratory distillation of even 4 litres of tar at a time. This quantity, however, we can and do distil in the laboratory of the Owens College by the method I have recommended, in thin glass retorts, and with perfect safety and ease, but I should prefer a distillation from a 10-gallon still, for in our case, using only 4 litres say, of normal coal tar, when the small quantity of crude benzol we obtain is treated with acid and alkali and rectified, a further classification into 90 per cent. and other grade benzols becomes nearly impracticable, the quantity has so dwindled down. In order to get the percentage of 0.75 of real anthracene, in the Simon-Carvès tar, *I did not include* the stuff obtained by coking the pitch, and found by Versmann to be so very impure. What I analysed was a perfectly normal and workable "thick oil," only it contained less liquid oils than ordinary anthracene oil does—in fact, it precisely resembled the best anthracene oils, after having been on the filters some time. With regard to Mr. Allen's suggestion as to heating petroleum oils strongly, I may just say that the processes originated by Liebermann and Burg and Letny, and afterwards carried out in South Russia, near Baku, for obtaining anthracene, etc., by passing Caucasian petroleum residues through red-hot tubes, were given up. The fact is, all methods based on the passing of oil vapours through red-hot tubes are exceedingly wasteful, owing to the incomplete nature of the decomposition to yield any one specific substance, and the large separations of carbon taking place. It is very interesting to hear that a chemical investigator has obtained a small quantity of a new hydrocarbon by such work with red-hot tubes, but to prepare such a hydrocarbon on a manufacturing scale would be to the manufacturer neither profitable nor interesting.

method, another reaction comes into play. The same is true of my process of titrating oils, which, indeed, is the case referred to by Dr. Armstrong, who appears to have overlooked the fact that an excess of hydrochloric acid is always present, and hence it is not a solution of a hypobromite which is in question at all, but the products of its decomposition by hydrochloric acid. If the solution of bromine in soda be freshly made, the reaction with hydrochloric acid may be represented by the equation—



Now, if the solution of bromine in soda be stale, a more or less perfect conversion of the hypobromite into bromate and bromide will have occurred, and hence in old solutions the action of hydrochloric acid may be formulated as follows:—



Hence, theoretically, the whole amount of bromine present in the liquid will be liberated on adding excess of hydrochloric acid, and it becomes a matter of indifference whether the bromised soda be fresh or stale. This fact has been clearly recognised by the compilers of the new German Pharmacopœia, who prescribe the employment of standard solutions of bromide and bromate of potassium in conjunction with acid for the titration of phenol.

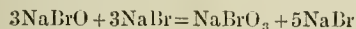
The facility with which the reaction resulting in the liberation of bromine will occur, and the completeness of the change in the cold may be open to question, and I have therefore made some experiments on the subject.

The solutions of hypobromite were made by dissolving bromine in excess of cold solution of caustic soda. The strength of each solution was then ascertained by measuring out 5c.c. with a pipette, diluting with water, adding potassium iodide solution and excess of hydrochloric acid, and titrating the liberated iodine by decinormal solution of thiosulphate. Operating in this way with three solutions of bromised soda I obtained the following results, calculated on 100c.c. of the solution:—

NOTE ON THE STABILITY OF HYPOBRO-  
MITE SOLUTION, AND ITS USE FOR THE  
TITRATION OF OILS, ETC.

BY ALFRED H. ALLEN.

THE reaction of bromine on caustic soda is well known to result in the formation of hypobromite and bromide of sodium, in accordance with the equation  $2\text{NaHO} + \text{Br}_2 = \text{NaBrO} + \text{NaBr} + \text{H}_2\text{O}$ . Gradually in the cold, but rapidly at a boiling temperature, a further change occurs with formation of bromate and bromide, thus—



Owing to this tendency to rearrangement, hypobromite solutions are generally described as quickly changing in strength, and Dr. Armstrong expressed this view in discussing the recent paper by Messrs. Mills and Snodgrass, "On the Quantitative Estimation of Oils and Fats."\* Of course such a change will be important if the hypobromite be used as such in the presence of excess of free alkali, as is done in estimating urea; but when the hypobromite solution is employed in conjunction with excess of hydrochloric acid, as in the assay of carbolic acid by Koppeschaar's

Bromised Soda.	Grammes of available Bromine per 100c.c.			
	Solution A. Solution B.		Solution C.	
Freshly made, contained ..	12.98	..	13.18	.. 12.37
Boiled 5 minutes without dilution ..	12.92	..	13.18	.. —
Diluted and boiled 5 minutes ..	12.94	..	12.88	.. —
Diluted and boiled 1 hour .....	12.80	..	12.64	.. —
Diluted and boiled 3 hours .....	12.60	..	—	.. —
Diluted and boiled 4 hours .....	—	..	12.56	.. —
Kept 3 weeks, contained .....	—	..	13.12	.. —
Kept 8 weeks, contained .....	—	..	—	.. 12.46
Kept 8 weeks and boiled 15 mins.	—	..	—	.. 11.65

Most of these experiments were made in duplicate. The three solutions of bromised soda presented a notable difference in colour, A being much lighter than B and C, though in each case a fair excess of soda was present. I am at a loss to account for the bright yellow colour of solutions of bromised soda, but it appears to bear a relation to the tendency to lose available bromine by boiling. The colour often becomes deeper by keeping, so that it can scarcely be due to hypobromite.

In the foregoing experiments the caustic soda employed was not weighed, but in the case of a fresh solution (D) 62.20 grms. weight of bromine was dissolved in a solution of 45 grms. of pure caustic soda made from sodium, which is a considerable excess, 31.1 grms. of NaHO being the quantity theoretically required for the reaction  $2\text{NaHO} + \text{Br}_2 = \text{NaBr} + \text{NaBrO} + \text{H}_2\text{O}$ . To insure great accuracy in the experiments with this solution 25 c.c. measure was diluted to 250 c.c., and 25 c.c. of the diluted liquid used for each titration. This volume required about



40c.c. of decinormal thiosulphate. The following results were obtained :—

(D) Bromised Soda—	Available Bromine in 100c.c.
	Grms.
Freshly made, contained (two experiments) . . .	12.86
Boiled for 1 hour, contained . . . . .	12.79
Boiled for 1 hour, contained . . . . .	12.76
Boiled for 4 hours, contained . . . . .	12.80

In this case the strength remained practically unchanged even after prolonged boiling, and yet the original solution was decidedly yellow. Hypobromite in solution is supposed to be readily and completely changed by boiling into a mixture of bromate and bromide, and the foregoing experiments were conducted on this assumption. As the result of direct experiment, however, I subsequently found that the solutions, after dilution and boiling for one hour, still gave a copious dark brown precipitate with manganese sulphate, and evolved gas when warmed with urea. Hence the hypobromite in a strongly alkaline solution of bromised soda is not readily destroyed by boiling. To ascertain whether this unexpected stability was due to the presence of caustic soda, this was removed from a quantity of hypobromite solution by adding an excess of magnesium sulphate solution and filtering the liquid. The liquid thus neutralised still had an alkaline reaction to litmus, but had a marked bromous odour which the original had not. It gave an abundant dark brown precipitate with sulphate of manganese, but five minutes' boiling usually sufficed to destroy every trace of hypobromite, so that hypobromite is readily and completely transformed by boiling in neutral solution. It must be borne in mind, however, that these experiments were practically made on hypobromite of magnesium, where, as in the former experiments, the sodium salt was employed. On boiling, the neutralised solution evolves a very notable proportion of bromine or compound of bromine capable of liberating iodine from potassium iodide, but the free bromine in the distillate, together with the available bromine in the liquid remaining in the flask, do not together amount to the full proportion of bromine contained in the solution before boiling.\* Still, the deficiency is not very great.

On the whole, therefore, it appears (1) that the presence of excess of caustic soda greatly increases the stability of hypobromite solutions. (2) That most solutions of bromised soda lose a little of their available bromine by boiling. (3) That the solution of bromine in excess of caustic soda solution has, after boiling, all the practical advantages which would be possessed by a concentrated solution of free bromine not liable to loss by volatilisation or change in strength by keeping.

A curious oversight of the advantages attaching to a solution of bromised soda over mere bromine water occurs in the reply of Messrs. Mills and Snodgrass to the discussion on their paper already referred to. These chemists describe the employment of hypobromite solution, together with excess of hydrochloric acid, as "an astonishingly roundabout way of preparing bromine water;" but the fact is that the use of hypobromite and acid affords a ready means of obtaining the necessary bromine in presence of a far smaller quantity of aqueous liquid than is the case if mere bromine water be employed. Thus nearly 300c.c. of a standardised solution of bromine water would be necessary for titrating 5c.c. of oil of turpentine, the solubility of bromine in water being only

about 3 per cent., whereas the solution of hypobromite I usually employ contains 12 per cent. of bromine, and can readily be made much stronger. Hence a much smaller volume is required for the titration, and the standard solution is not liable to lose strength by exposure to air, as would be the case with mere bromine water. In their reply to the discussion on their paper (page 438 of last volume), Messrs. Mills and Snodgrass state that "the bromine-water method or moist process was communicated by one of us to Mr. Allen in 1880 (Oct. 7th)." There is a mistake here, the origin of which I am wholly unable to trace; but, as a matter of fact, I had given a description of the results yielded by the process in a paper read before the Pharmaceutical Conference in August, 1880, and published in the *Pharmaceutical Journal* for September 25th, 1880, which latter date was twelve days before the communication is stated to have been made to me\* (October 7th, 1880).

The method I have employed for preparing the solution of bromised soda has incidentally furnished a determination of the density of bromine. The plan adopted has been as follows: About 40 to 45grms. weight of pure caustic soda were dissolved in water, and the solution was placed in a flask and accurately weighed. A quantity of bromine was shaken with powdered potassium bromide, water added, and the shaking repeated. The bromine was drawn off from the aqueous liquid, shaken with water, separated, and then agitated with concentrated sulphuric acid. By this treatment it was hoped to purify the bromine from any chlorine it might have contained, and also to render it perfectly anhydrous. The purified bromine was next run into a burette, and from that 20c.c. measure was run into the soda solution, and the increase of weight noted. In this way the weight of 20c.c. of bromine at the ordinary temperature of the laboratory was accurately ascertained, and in three experiments was found to be 62.261, 62.381, and 62.202grms., the mean being 62.281. This would correspond to a density of 3.114 if the weight of 20c.c. of water at the ordinary temperature had been strictly 20grms., but with a correction based on the actual weight of water delivered by the burette, the density of the bromine at the ordinary temperature works out to 3.177. I do not claim rigid accuracy for this determination, but it is of interest as approximately confirming the result of Pierre (3.1872 at 0°C.), while it is at variance with the figures of Balard (2.966) and Löwig (2.98 to 2.99 at 15°C.).

There is one curious fact in connection with these experiments for which I am unable to offer any explanation of a satisfactory nature, and that is that in each case the proportion of bromine found in the bromised soda by titration is sensibly in excess of the weight actually employed. Thus:—

	A.	B.	D.
Bromine employed, by weight . . .	12.432	12.929	12.440
Bromine found, by titration . . . .	12.98	13.18	12.86

As to the cause of this discrepancy, (1) the precautions taken to purify the bromine used in making solutions A and D seem to negative the presence of chlorine as an impurity; (2) in each case the thiosulphate solution employed for the titration was most carefully set by resublimed iodine; (3) the potassium

\* The theoretical bearings of the decomposition of magnesium hypobromite with evolution of more or less bromine, and the quantitative experiments required for the elucidation of the reaction, I propose to make the subject for a future paper.

\* The following is a passage which occurs in the paper mentioned in the text: "Those products which are most readily acted on by nitric acid also have the greatest capacity for decolorising bromine water, and I am attempting to base a method of determining the olefines on this principle. The results already obtained in this manner are very remarkable, and conclusively prove the predominance of olefines in shale products, these readily decolorising many times the volume of bromine which can be caused to combine with petroleum naphtha or burning oil."

iodide employed was free from iodate; (4) any inactive impurity in the bromine, such as bromoform, bromide of carbon, water, or sulphuric acid, would tend to reduce its titration-value as compared with its weight; (5) the presence of iodine would operate in the same direction.

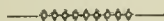
#### DISCUSSION ON MR. ALLEN'S PAPER.

MR. WATSON SMITH: A stable solution of sodium hypobromite of known strength means an available bromine solution of known strength ready at any moment. Such a solution may in the future furnish a useful means for titrating certain naphthas (*e.g.*, shale spirits) for their content of olefines, adding the bromine so long as quickly absorbed without formation of hydrobromic acid. A method based on this principle has, I believe, already been foreshadowed by Mr. Allen.

MR. ALLEN said he had described a method of employing bromised soda and hydrochloric acid for the titration of shale-products, in his work on "Commercial Organic Analysis."

DR. CAMPELL BROWN thought that a new determination was wanted of the atomic weight of bromine.

DR. HURTER said that the loss of strength alluded to in the paper was also found in boiling the hypochlorites. They found there was not the amount of chlorate present that would correspond to the original amount, and they always put it down to simple escape of oxygen. He confessed himself quite unable to account for the loss in weight Mr. Allen had spoken of.



### AN ATTEMPT TO UTILISE WASTE HEAT BY PERKIN'S PIPES.

BY FERDINAND HURTER, PH.D.

THE utilisation of waste heat forms the subject of many inventions and of many disappointing experiences. To save heat does not always mean a saving of money. An apparatus which pays at one place does not pay in another. Whether the utilisation of waste heat is equivalent to a saving of money depends entirely upon the work which has to be done and upon the price of coal. It is impossible to settle the value of waste heat generally. But for particular applications its cost can be ascertained—for instance, for its application to the evaporation of liquids; and it will be found that for this purpose waste heat will not often mean a great saving of money. Assuming that the waste heat itself has no value, its cost will be the cost of the apparatus by means of which we utilise it. Supposing that depreciation, repairs, rates, and taxes, ground rent, supervision, etc., are covered by the modest sum of 20 per cent. per annum on the capital outlay, the cost of waste heat per annum would be one-fifth of capital employed in utilising it. Every square foot of heating surface of sheet iron transmits per hour as much heat as would heat 4 lb. of water by 1° C. where there is 1° difference of temperature between the two sides of the heating surface; consequently per year of 300 working days that square foot of heating surface would transmit 3.12 ton units of heat (1 ton unit equals as much heat as would raise the temperature of 1 ton of water by 1° C). But if the difference of temperature were any other number, say  $T$  degrees, then the number of ton units transmitted per annum would be  $3.12 \times T$ . Now, this quantity of heat costs one-fifth of the capital spent upon one square foot of the heating surface. The least amount of money for which any apparatus

for evaporation per square foot can be erected is about 4s. to 5s., including mounting, brickwork, etc., consequently the cost of  $3.12 \times T$  ton units of heat costs from 9½d. to 1s.

Let us now see what in actual practice 1 ton unit of heat direct from coal costs us when applied to evaporation of water in a boiler.

Supposing that a good boiler, capable of evaporating 36 cubic feet of water, costs us £400, and that it evaporates per annum of 300 days 7,200 tons of water, using 1,200 tons of coal, we have the following annual charges:—

Interest, etc. (20%).....	£80
Coal at 5s.....	300
Man for firing .....	25
	£105=97,200d.

The evaporation of 7,200 tons of water requires 3,859,200 ton units of heat. Thus, 1 ton unit costs us nearly one-fortieth of a penny. If, therefore, 1 ton unit of heat gained from waste heat must not cost more than 1 ton unit of heat gained directly from coal, it is necessary that the temperature of the waste heat be such that the fraction

$$\frac{9\frac{1}{2}d.}{3.12 T} = \frac{1}{40}d. \text{ or that } T = \frac{40 \times 9.5}{3.12} = 120^\circ \text{ C.}$$

This fraction shows that unless the difference of temperature between the two sides of the heating surface be at least 120° C., the waste heat will cost more than the heat from coal direct, if that can be obtained at 5s. a ton, of such quality that one ton will evaporate six tons of water in an ordinary boiler with ordinary firing. Thus in this country the utilisation of waste heat, if it must be a saving of money, requires that the fuel gases from which this heat must be abstracted shall be at a temperature of at least 220° C. or 428° F. You will perceive that if the temperature approaches this the money saved by utilising the heat is a very small amount indeed. This limit, beyond which the utilisation of waste heat costs more than the heat is worth, rises as the cost of the apparatus rises. Where coal is more expensive the utilisation of waste heat of a lower degree of temperature is a feasible undertaking. If I now say that per 1 ft. heating area Perkin's water-heating apparatus, as applied to evaporation, costs 8s., you will readily perceive that there is no great inducement to try that system, unless the temperature of the waste heat be more than 800° F., or local circumstances render it specially favourable as compared with other apparatus.

In Leblanc soda works there is a great amount of waste heat at disposal, and in Messrs. Gaskell, Deacon, and Company's works, where special processes are in use, the question arose how to utilise the waste heat of a revolving black-ash furnace. The hot gas from the revolver was first passed through the internal flue of a large boiler, and then by means of a flue of 12 ft. width and 3 ft. depth under a wrought-iron concentrating pan 36 ft. in length. The total amount of heating surface thus disposed for the utilisation of waste heat was 830 square feet. But the quantity and intensity of waste heat from even small revolving furnaces is such that these 830 square feet were inadequate and only reduced the temperature of the fuel gases to about 1,000° F. It was therefore desirable to increase the heating surface, but owing to the already inconveniently crowded state of this part of the works it was impossible to simply add the cheapest form of evaporating apparatus, an iron pan. Consequently it was decided to enlarge the heating surface by placing into the flue, under the pan, a series of coils of pipes on Perkin's high-pressure system. Design and execution were left to an



engineer experienced in constructions of this kind. After mature consideration the following arrangement was decided to be carried out: Six separate systems of coils were used. Each system consisted of two coils, one placed into the flue under the pan the other into the pan. The length of the coil in the fire was 160ft., that in the pan 70ft. The aggregate length of all the coils was 1,380ft., and the heating area 360 square feet. Each coil was provided at its highest place with an expansion vessel, and at its lowest point with a kind of ball valve, through which the pipes were filled with water. The pipes were very strong,  $1\frac{1}{2}$ in. outside and  $\frac{3}{4}$ in. inside diameter. The joints were made as usual in this kind of apparatus, the end of one pipe being sharpened to a knife edge and hardened, meeting the flat and soft end of the pipe to which it was to be joined, the one pipe having a left-handed and the other a right-handed thread. The two pipes were screwed together by a coupling, which had corresponding threads, and by means of very powerful tools the knife edge was driven into the flat end of the pipe to be joined. The six coils in the flue were placed across the flue supported by suitable frames. Opposite each coil was a door to clean the pipes from flue dust. The coils in the pan were placed at the bottom of it lengthwise. Each system contained, when full, about 1 cubic foot of water, and it was calculated that this water, in circulating through the coils, should reach a temperature not exceeding  $450^{\circ}$  F., and the expansion vessels were so adjusted that they would allow this cube foot of water to expand. But when the apparatus was started it was very soon found that the water became much hotter than was anticipated. The practical way of ascertaining the temperature consists in filing, at the place at which the hot water leaves the flue and enters the pan, a bright surface on the pipe, and observing the colour which the bright spot assumes. It ought to become straw coloured. But in our case the colour rapidly became purple and blue, and thus all we could ascertain was that the temperature exceeded  $550^{\circ}$ . The first of the coils was also provided with a pressure gauge, but as the index very rapidly moved to the end of the scale 1,500lb. per square inch, we had no means of judging the temperature. The expansion vessels proved too small, and the first and second of the coils, those exposed to the greatest heat, burst during the next twelve hours. According to Hirn's formula, if water is heated to  $300^{\circ}$  C. ( $572^{\circ}$  F.) it will expand nearly 40 per cent. of its volume, from which the expansion of the iron apparatus would have to be deducted. These expansion vessels are a weak spot in the whole apparatus. If they are too large the pressure is too small and the water boils; if they are too small the pressure becomes too large and the pipes burst. When the apparatus had been provided with expansion vessels of sufficient capacity it worked, but it did not work well. The temperature outside the pipes being variable interfered much with the work of the pipes. At times the fluid in the pipes became actually red hot, to judge from the red heat of the pipes which conveyed it from the coils in the flue to the coils in the pan, and which were quite outside the flue. Very soon the pipes became covered with flue dust, and this interfered very seriously with the amount of heat conveyed. During one experiment I found that instead of conveying 4lb. units per hour they only conveyed about 2lb. units, and probably less. The dust was frequently blown off by means of a steam jet. Then the number of pipes in the flue proved a serious hindrance to the draught. Unless very good slack was at our disposal the revolving furnace got behind in its work. But the most serious drawback was, that although the workmanship was good the coils leaked and gradually got empty; and as they cannot

be refilled except when cold, this leakage meant so serious a stoppage that the pipes had to be removed. This failure, however, is chiefly due to the following cause: The water circulates in the pipes by reason of the difference of the specific gravity of the hot water ascending and the cold water descending. The velocity of circulation depends upon the difference of level between the coils. But it also depends upon the length of the system and the number of bends, and it requires a very careful calculation to find the length of the pipes for any given temperature and any given difference of level. A method of making this calculation is given by Schinz in *Dingler's Journal*, vol. 219, but it is so complicated that I must condemn it as not applicable. I have felt that, failure as it was in our case, the application of this system might still have a future in the utilisation of waste heat; and in order to know the probable cause of the failure, I have taken the trouble to carefully construct formula for the ready calculation of such pipes. I find that in our case the failure was mainly due to too slow a circulation. The difference of level from centre to centre of coil being only 1ft. 6in., the total length of the pipes in each coil ought not to have exceeded 144ft., whereas we had 230ft. Thus we had far too much friction. But the slow circulation in too long a pipe causes a high temperature, a high pressure, and consequently increased leakage and danger to the apparatus.

I shall append tables for readily calculating such systems of pipes. It may be interesting to some to know how far the heat evolved in the fireplace of the revolver was thus utilised. The following figures are approximations as good as I could obtain them. There were burnt per week 120 tons of fuel, including mixing slack, and 114 tons of sodium sulphate were decomposed.

	Ton Units per Week.
Heat produced in fireplace of revolver.....	840,000
Heat utilised in revolver: (a) For decomposition of sodium sulphate and calcium carbonate into sodium carbonate and calcium sulphide.....	188,100
(b) Carried off by fused black ash.....	38,760
	226,860
Heat utilised for evaporation: (a) In boiler (area 396 square feet).....	89,190
(b) In pan (area of bottom, 432 square feet).....	51,990
Assisted by coil area, 360 square feet.....	141,180
Heat lost: (a) By chimney (gas at $800^{\circ}$ F.).....	138,624
(b) Other losses (difference).....	333,720
	472,344
	840,000

When the same revolver was working in the ordinary way—i.e., when its waste heat was utilised for evaporation of vat liquor by surface evaporation—the following figures were similarly computed:—

Heat evolved in fireplace .....	840,000
Utilised in revolver .....	226,860
Utilised in evaporation of vat liquor by 432 square feet area .....	138,624

The two statements compare as follows:—

Percentage of heat utilised by direct surface evaporation, heat passing over surface of liquid, 436 square feet area.....	16.5
Percentage of heat utilised by 1,188 square feet of heating surface, heat transmitted through iron plates ..	16.8
Ton units per square foot of area of liquid exposed to surface evaporation per week.....	320
Apparatus cost per square foot .....	5s.
Ton units per square foot of surface of transmission per week.....	119
A square foot costs .....	9s.

The conclusion is obvious: the cheapest way of utilising waste heat is by surface evaporation, where that is practicable.

The formula I have constructed for the ready calcu

lation of apparatus similar to the one described is, if we denote by—

H = Heating surface of the single coil in the fire.  
C = Cooling surface of the coil placed into the liquid.  
 $\alpha$  = 23 Kilogramme units of heat transmitted per square metre per degree per hour from the fire to the water.  
 $\beta$  = 921 Kilogramme units transmitted per square metre per degree difference from hot pipes to the liquids in evaporating pan.  
 $T_1$  = Temperature of fuel gas.  
 $t_1$  = Highest temperature of the circulating water.  
 $t_0$  = Lowest temperature of water in pipes.  
 $T_0$  = Boiling point of the liquid in the pan.

$$\delta = \frac{H}{C} = \frac{\beta}{\alpha} \cdot \frac{\log \frac{T_1 - t_0}{T_1 - t_1}}{\log \frac{t_1 - T_0}{t_0 - T_0}}$$

The following table gives the different ratios of heating surface to cooling surface which must be used for different temperatures.

$T_1$ Centigrade.	$T_1$ Fahrenheit.	$\delta$ Ratio $\frac{H}{C}$
1000	1832	3.018
900	1652	3.132
800	1472	3.990
700	1292	4.756
600	1112	5.913
500	932	7.801
400	752	11.515

To obtain values for the absolute lengths of the coils, I have assumed the boiling point of the liquid to be 100° C. = 212° F., the lowest temperature of the circulating water 120° C. = 248° F., and the highest admissible temperature 240° C. = 464° F., corresponding to a pressure of from 500lb. to 600lb. per square inch. I have also taken into consideration the friction of the water in the pipes, but not the bends. Consequently the following values are maxima, and must on no account be surpassed. The formula for the length of the pipes to be placed into the evaporating apparatus is, if D is the diameter of the pipe in inches,

$$C = 37.3 \times D \sqrt{\frac{h}{1 + \delta}}$$

In this formula  $\delta$  has the value given in Table I., and  $h$  is the difference of level in feet between the centres of the coils in the fire and those in the evaporating apparatus. The following table gives a few of these values.

TABLE II.

Showing length in feet of pipes necessary for one unit of Perkin's system (diameter 1in.).

Difference of Level Feet.		1000° C.	900° C.	800° C.	700° C.	600° C.	500° C.	400° C.
2	C	29.5	28.6	27.5	26.5	24.5	22.7	20.0
	H	83.0	98.0	110	126	144	177	230
4	C	37.5	36.4	34.9	33.5	31.2	28.7	25.5
	H	112	124	139	153	181	224	290
6	C	43	41.5	39.8	38.1	35.5	33.0	29.0
	H	128	142	157	180	208	257	333
8	C	47.1	45.7	44	42	39.3	36.5	32.1
	H	141	156	176	198	231	282	372
10	C	50.7	49.1	47.5	45.5	42.5	39.1	34.5
	H	152	168	188	215	252	305	395

Example: Difference of level between centres of coils equals 8ft.; temperature of fire, 800° C. The table gives opposite 8 and under 800° the two figures 44 and 176, which mean that 176ft. length of coil must be placed into the flue, and 44ft. into the liquid to be evaporated. The water in the pipes is supposed

to have as extreme temperatures 120° C. and 240°, and its volume will be about 1.059 at 120°, and 1.262 at 240°, if the volume at 0° = 1, consequently the expansion vessels must contain 25 per cent. of the volume of the pipes.

## DISCUSSION ON DR. HURTER'S PAPER.

Dr. CAMPBELL BROWN asked what happened when the tubes burst. He supposed the expansion of the water was so small that the explosion could be of very little effect.

The CHAIRMAN asked whether there was not a deposit on the tubes from the alkaline liquor above them which would obstruct the heat.

Dr. HURTER said the liquor was not so saturated as to deposit salts, and consequently the pipes remained clean. The expansion of water at such degrees of heat would be very great indeed. At 300° C. water was expanded 40 per cent. beyond its natural volume, but in the case of the bursting of a pipe there was very little damage indeed.

Dr. BROWN: No great volume of steam?

Dr. HURTER: Very little. The whole of one of these systems I have described contains about one cubic foot of water.

The CHAIRMAN thought the fact of having tubes filled with red-hot water would be new to most of them, and Dr. Hurter had seen something which few people had seen before.

## Manchester Section.

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Vice-Chairman: George E. Davis, F.I.C., F.C.S.

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C. Estcourt.  
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J. von Hohenhausen.

J. Jackson.  
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C. Schofield.  
R. Angus Smith.  
W. Vickers.  
D. Watson.

## Hon. Sec.:

J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

MEETING TUESDAY, NOVEMBER 6TH, 1883.

## ADDRESS BY THE CHAIRMAN,

I. LEVINSTEIN, ESQ.

THIS Section has been formed for the purpose of more effectually carrying out the principal object of the foundation of our Society,—namely, the promotion and advancement of the knowledge of technical chemistry. That our Society has been thus far successful is generally admitted, but there is still plenty of scope for further improvement and plenty of work to do, and I hope that the Manchester Section, so favourably situated, will lead the way in promoting its principal aims. Manchester, as has been frequently pointed out, is an important chemical centre, many most extensive chemical manufactories being situated within a radius of less than twenty-five miles. We find in its neighbourhood large alkali, soap, acid, and artificial manure works, extensive tar distilleries, aniline oil and coal-tar colour works, etc. Indeed, nearly every branch of chemical manufacture is represented; and if we further take into consideration the numerous important cotton and other industries in which chemical knowledge plays such an essential part—such as printing, dyeing, sizing, bleaching, paper-making, etc.—we may fairly say that Manchester, above all, is the very town where the establishment of a Section of our Society was most desirable; and this was undoubtedly



the opinion also of those who, about nine months ago, bestirred themselves so energetically for its formation. Now, gentlemen, notwithstanding that the prosperity of a manufacturing nation depends to a great extent on the progress of its chemical and other allied industries, we frequently hear from men who ought to be well informed that this country is behind others, not only in the knowledge but in the application of chemistry, which in the case of a great manufacturing nation cannot fail, if it be a fact, to be an extremely disheartening reflection.

We will now consider how far the serious statement relative to our being behindhand in the application of chemical knowledge is correct. If we take into account the very large capital invested in chemical industries, and the very important bearing of the latter on other industries, it will be conceded that we in England ought to watch with the greatest jealousy the progress of other nations, and to examine carefully and apart from prejudice their position as compared with our own. Now as far as any decline or falling behind in chemical industries is concerned it may be some consolation to know that this is principally confined to one special branch, and that the assertion, frequently heard, that we are completely beaten in those industries which depend to a large extent on applied chemistry is as much an exaggeration as would be the assertion that the French can beat the British on the seas. We are often told that the English calico printers cannot print so well as the Alsatians, that our dyers cannot dye so well as the French or Germans; but such indiscriminate statements can be accepted only to a very limited extent. If an outsider, as sometimes happens, picks up one of the very best class Alsatian prints, and compares it, without taking into account the relative cost, with an average English print, and then bases his criticism on such a comparison, he may easily jump to the conclusion that this country is behind others as regards printing. English and Scotch printers and dyers can print and dye, taking price and quality into consideration, equally as good colours as the Continental, and indeed in some specialities they are ahead of any, even the foremost.

We must never overlook the fact that the demand regulates not only the quantity but also the quality of the supply, and I doubt not that if our merchants were willing to pay our printers as good a price as the highest class of Alsatian prints command, and would at the same time undertake to keep their works fully going with orders, the English printers would be able to turn out as good a print as any one. It is at any rate a fact, and one which tests the standard of an industry more conclusively than all the loud assertions about its failings, that the printers of this country supply not alone the principal part of our home consumption but also to some extent the wants of other nations, and that in some classes of prints they are still taking the lead in those foreign markets where prohibitive tariffs do not exclude fair competition. And very much the same thing may be said about the position of the art of dyeing. It is quite true that the Swiss and others produce, on certain classes of goods, rather better colours, but here again it is in many cases a question of price. I know by experience that English dyers can produce far superior colours to those generally met with in the trade, if the merchant could only afford a better price for dyeing; but the prices which dyers have to accept for some colours are so exceedingly low, considering the high wages they have to pay for labour, that it becomes an impossibility to supply the very finest shades. What is generally asked of the English dyer is to turn out quantities at the smallest possible expense consistent with producing passable colours.

So here again it is the demand which regulates the quality of the supply; and in justice to the dyers of this country I must mention that for certain classes of goods even the Germans prefer them to their own dyers, whilst some of the principal concerns in the United States employ Scotchmen and Englishmen as foremen dyers in their establishments.

That this country is foremost in such industries as the manufacture of alkalis, soap, acids, artificial manures, bleaching powder, paper, etc., is also evident, and is proved by the extensive export trade in these different products. The fact that some classes of paper are imported is due to cheaper raw material and cheaper labour, but as far as quality is concerned our high-class writing, news, tissue, and drawing papers rank certainly among the very best produced. We may therefore say, gentlemen, without boasting, that England still occupies a very prominent position in many branches of chemical industry and the manufactures depending on it; and it is only that special part which deals with the production of aromatic hydrocarbon derivatives that has been neglected, and in which this country has allowed itself to remain behind others. This industry, of comparatively recent origin, has already become one of the most important, for not only has it revolutionised the whole art of dyeing and printing, by the introduction of the aniline, naphthol, alizarin, and resorcine colours, but it includes also the production of numerous other compounds of more or less utility to the arts and manufactures and to science. I will only just mention artificial vanilline, artificial oil of bitter almonds (benzaldehyde), carbolic acid, salicylic acid, and kairin, one of the latest discoveries, which promises to be of great service in medicine. The establishment and development of this industry was due to the careful study of the constitution of the aromatic hydrocarbons and their derivatives, a group of chemicals which has supplied the chemist with a wider field for original research, and one followed by more important practical results than most others, and in which are yet hidden the germs of perhaps still more important discoveries. The great commercial importance of this industry may be estimated by the amount of capital invested in it. The yearly turnover represents several millions of pounds, with profits amounting to many hundred thousands (the profits of three large German coal-tar colour companies alone reached last year a million sterling), and we must not forget that this industry is as yet comparatively in its infancy, and that its further development may multiply these amounts many times over.

If we carefully observe the constant improvements which are being made by the Germans in the manufacture of artificial indigo, and at the same time keep in mind the probable great reduction in the price of the aromatic hydrocarbons, such as benzene and toluene, we shall come to the conclusion that its commercial success as a manufactured article and its general introduction to the trade will only be a question of time. If we further consider the recent researches by Skraup, Königs, Fischer, Ladenburg, and others, it is not at all improbable that we may any day be startled by the news that the German savants have succeeded in producing, at a price low enough to compete not alone with artificial indigo but also quinine and the alkaloids of opium, or at least their physiological equivalents. This feat would mean almost incalculable losses to some of the most important colonies of this country.

It would take us too far from our subject to refer to the physiological properties of some of the compounds already produced from the aromatic bases, but it may perhaps interest you to know that the commercial production of one of the most important—that is kairin—is extending. When this substance was first

discovered, and its effects on the human system recognised, it was then thought that it would probably entirely replace quinine.\* You will thus see, gentlemen, that the artificial production of these most valuable and important articles of commerce, hitherto obtainable only through the chemistry of nature, is not so very remote.

It will therefore be self-evident that a sound knowledge of the constitution of the aromatic hydrocarbons and their derivatives is becoming every day of more importance to the technical chemist, and it is unfortunate for this country that just in this very department of chemical knowledge the British chemist has been entirely outflanked by the Germans, who have made the study a speciality, and during the last twenty years have cultivated and developed it in a marvellous degree. You will also agree with me that it is a perfect anomaly that England, the largest manufacturing nation, possessing immense accumulated wealth, with general manufacturing facilities superior to those of any other country, situated geographically in the most favoured position, and, above all, possessing the necessary raw material in quantities sufficient to supply both its own wants and the needs of the world, should be altogether beaten in an industry which had its original development in its own borders, and of which it was indeed the founder. What, then, is the cause of the fact that this country has lost and is still losing ground as regards this special industry? Is it really the want or the insufficiency of our technical schools? I for one must confess that I cannot see how technical schools or an improved general technical education can have much to do with an industry requiring the highest scientific knowledge. Do not misunderstand me. I do not say that the technical schools in this country are as numerous or as perfect as is desirable. On the contrary, I quite agree that a better equipment and an increase of the number of such schools, and, above all, a general reorganisation of the present system of teaching inductive sciences, would prove a great boon to the general industries of this country. What I contend for is that these schools have only an indirect bearing on the question of this country's position with regard to the special industry which is the principal subject of this evening's address.

Only consider, gentlemen, that about twenty-five years ago England founded this industry, and for a number of years maintained her supremacy in it at a time when the educational system and the number of technical schools were far inferior to what they are to-day; and further, that this country has made comparatively far quicker strides in general education and in the foundation of new schools within the last fifteen years than Germany—and yet, notwithstanding all this, the latter has taken this industry out of our hands. The reason of the inferior position at present occupied by England with regard to this industry is not as mysterious as it might at first sight appear. We will now endeavour to trace the cause of this singular deficiency, which in my opinion amounts to nothing less than a national calamity. If in doing so I have to refer to certain matters which may be unpleasant for some, I trust that the vast importance of the subject will plead my excuse, if any is needed. I may, however, assure those who may differ from me that it is very far from my desire to make any frivolous charges, and that my practical and scientific experience, extending over a period of twenty years, as a manufacturer of coal-tar colours and other aromatic hydro-

carbon derivatives, employing a number of English and foreign scientific chemists, has perhaps afforded me special facilities and opportunities of forming, to some extent, a sound opinion as to the reasons of England's deficiencies in this particular branch of scientific knowledge. However, before commencing the consideration of the real cause of this deficiency, I would ask you to carry your minds back to about twenty-five years ago, when Dr. A. W. Hofmann (whose labours in this special department of organic chemistry had already made him famous) was professor at the Royal College of Chemistry. This retrospect will serve two purposes—it will show you, first, the proud position which this country once occupied with regard to this ever-extending industry; and secondly, the very great influence which one eminent teacher exercised in its foundation, and still more in its development. It was to Hofmann that was due the fresh impetus given in this country to the study of organic chemistry. Not only was he himself constantly engaged in original and laborious researches but he possessed also the talent of inspiring his pupils with a like enthusiasm for that department of chemistry in which this eminent worker and teacher obtained an everlasting name. It was in Hofmann's time that one of his pupils, Mr. Perkin, discovered the first practical method of obtaining a colouring matter from aniline—a colouring matter known for some years as Perkin violet or mauve—and the credit of having founded the entire industry of the manufacture of coal-tar colours belongs to England, though it is quite true that Runge and others, long before Perkin, had observed that colouring matters might be produced from aniline and other hydrocarbon derivatives, but it was Perkin who first devised a practical method of obtaining such colours, and to him belongs also the honour of having first produced them on a manufacturing scale, and of having first introduced one of them to the trade.

It was in Hofmann's time that English chemists improved upon the then costly and difficult method of making aniline oil, and so well did they succeed in their efforts that even at the present day the same process, with very immaterial alterations in the apparatus, is still employed. To give you some idea of the estimation in which the skill of the English chemists in this special branch was at that time held by the Germans, I may remind you of a rather amusing episode, which may possibly be yet in the memory of some, when a partner in one of the largest German coal-tar colour companies fared rather roughly at the hands of British workmen, in consequence of his eagerness in trying to obtain, somewhat indiscreetly, information respecting the different processes carried on at that time at a certain works. It was also in Hofmann's time that another of his gifted pupils, Mr. E. C. Nicholson, in conjunction with Messrs. Maule and Simpson, erected the famous works at Hackney Wick. These works, which were carried on under the name of Simpson, Maule, and Nicholson, were for a number of years the most extensive coal-tar colour works in the world, and they and their successors not only supplied this country but also a considerable portion of the Continent and Transatlantic countries with their superior products; and it was at the same works, in connection with Mr. Barnes, that the manufacture of pure aniline, pure rosaniline and its salts, the phenylated rosanilines and their soluble compounds—such as soluble blues, cotton blues, Nicholson's alkali blues—phosphine, Hofmann's violets, etc., were some of them first produced and others brought to a degree of perfection which has not, even in the present day, been surpassed. Hofmann left this country about 1866. A few years

\* In justice to English chemists, I must here not omit to mention the meritorious work of Professor Dewar, on quinine, the basis of kairin, and also the valuable experiments which he made, in conjunction with McKendrick, in order to show the physiological properties of the quinoline and pyridine bases.



afterwards Perkin and Nicholson retired from the business of coal-tar colour makers, and from that time this branch of manufacture has remained comparatively stationary in this country.

The manufacture of aromatic hydrocarbon derivatives requires the highest special scientific chemical knowledge, as it consists of a series of chemical operations which are difficult even for a skilled chemist, who is not a specialist, to perform in the laboratory, and in which chemicals are constantly introduced which are often known only by name to the most experienced chemists, and even to them only as chemical curiosities. Further, in order to keep pace with the continual new improvements and discoveries chemists of the highest special knowledge, and able to strike out new lines, are a *conditio sine qua non*, if this industry is to be carried on successfully. The field of chemical science is so extended, and is constantly extending so rapidly, that it becomes simply an impossibility for any chemist to attain to eminence in every branch, and certainly impossible for a technical chemist to do so. The work must therefore be divided, and just as specialists are becoming more and more in request in medicine, and surgery, and other sciences, even more so are specialists necessary in theoretical as well as in technical chemistry, and it is to their eminent specialists that the success of the German chemists is to a great extent due. The German chemists, appreciating from its beginning the importance of this new industry, associated with capitalists, and ably supported by their eminent specialists and their large staff of pupils, with their minds actively at work in the same direction as their tutors, soon succeeded, after this country lost its only great teacher in this particular branch, in making themselves masters of the field. This really need not surprise us when we consider the extraordinary want of interest displayed in this special direction by the British professors. I do not mean to say that we have not eminent chemical teachers—the names of many will occur to you. But then these gentlemen, though even great organic chemists, are none of them specialists in this particular department of science, and if you desire further confirmation of my statements, I invite you to look through the literature of the last ten years, and to compare the researches and investigations undertaken by the German with those of the English professors bearing on this subject, when you will be quite as much astonished at the overwhelming amount done by the former as at the insignificance of the work done by the latter. Indeed, gentlemen, unless my memory plays me false, I do not remember, during the period referred to, with perhaps few exceptions, any important original research bearing on this subject undertaken by the English professors, and I certainly cannot call to mind any work done by them which has been of practical utility to this industry. That nothing great has been achieved by them in the direction referred to during the last twelve months may be fairly gathered from the presidential address and the papers read in the chemical section of the British Association at Southport;\* and whilst the Germans are constantly developing this department of chemistry, and are thus extending and cultivating year by year the ground taken from this country, the British are content with simply acknowledging the ever-increasing amount of experimental research in Germany, without making the slightest effort to overtake their opponents, save perhaps by the demand for increased expenditure on technical education or endowments for research.

Having thus endeavoured to trace the cause of the decline of the manufacture of the hydrocarbon derivatives the remedy cannot fail to recommend itself. What are wanted are teachers of the highest talent who have made this special department of chemical science their life-study, men like Perkin, Hofmann, Kekulé, Baeyer, Fischer, Victor Meyer, Graebe, Liebermann, Griess, etc., and I venture to assert, without assuming the rôle of a prophet, that with a few such teachers the average English chemist would very soon equal the German in this branch of knowledge, without incurring fresh expenditure for new technical schools and endowments for experimental research. There are at present quite sufficient schools to effect this purpose. We have in this country splendidly equipped chemical laboratories, munificently supported, but whether teachers or pupils are to blame, the results of the teaching of this branch are unsatisfactory. Men are wanted who have made this particular part of organic chemistry their own, and who could guide our young chemists to original work in this direction. Let the proper authorities select the most talented men as teachers. Let them follow the German authorities, who, when a professorship becomes vacant, look out anxiously for men who have distinguished themselves by some important original work, regardless of any other consideration. A striking illustration of this is found in the case of Messrs. Graebe and Liebermann, both of whom were chemists in a printworks at Berlin, when they discovered the process of producing artificial alizarin. Not long after the publication of their admirable work they were appointed lecturers and shortly after professors. Let the right men be appointed in the right places, and not only will this country be able once more to take the lead in an industry for which it possesses every natural advantage, but the arts of printing and dyeing will also be correspondingly benefited. With reference to these latter, I may say that thus far the experience of printers and dyers with the average chemists, trained at even the best British colleges, has not been very encouraging, and they still have to rely principally upon the much-abused rule-of-thumb man; for, after all, a man with experience gained during a lifetime in these trades, even though he possesses no knowledge of the science which has such an important bearing upon them, is still far more useful to dyers and printers than a mediocre average chemist; and this, I believe, is also the universal opinion of the dyers and printers of this country, who, where they do employ chemists, use them, as a rule, simply to test the quality of the drugs required in their business. Such a class of chemists will not do much to advance the progress of these industries. It needs also no great amount of clear-sightedness to perceive that if we can turn out of our universities and colleges not mere testers, but chemists with a superior knowledge of the hydrocarbon derivatives, and especially of the coal-tar colours, which have caused such a revolution in dyeing and printing, and are becoming daily of greater importance, such chemists will not only be far more useful in promoting the advancement of these arts but they will also readily find far more remunerative employment than our present chemists.

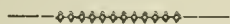
However, I do not mean to say, gentlemen, that if we appoint a really eminent specialist, he would retrieve all at once the lost position of this country. What I desire to impress upon you is that such an appointment would be the right step in the right direction. I am quite aware that even with the most eminent men as teachers it will take a few years before England will be able to outstrip the Germans in the race in which she has allowed them to get such

\* "Researches on Secondary and Tertiary Azo-compounds" (No. 2, *Jour. Chem. Society*, Nov., 1883. This paper was read by R. Meldola, the chemist in an English coal-tar colour factory.

a start. The existing coal-tar colour works in Germany are in the hands of powerful companies, employing large staffs of highly-trained chemists, supported by the teachers of the various college laboratories, who are further supported by a large number of competent assistants.

In the meantime let this Section do its utmost in promoting the advancement of that industry in which this country has at present unfortunately to deplore its deficiency. Other Sections of our Society are already most ably represented in the alkali and other manufactures; and I have great pleasure in informing you that some gentlemen have been kind enough to promise us for the ensuing session papers bearing on the subject with which, I am afraid, I have already occupied too much of your valuable time.

The CHAIRMAN, at the close of his address, said: I wish to make a few remarks as to the suitability of papers for our Section. Some gentlemen are under the impression that papers are only suitable containing communications of some new discovery or invention. I venture to think that all papers are most welcome contributions which embody the results of experiences gathered by the author in any special branch of industrial chemistry, or which deal with any new branch not fully developed in this country, and, finally, papers consisting of concise compilations, and supplemented by comments on the latest literature of new compounds, which are likely to be introduced into the chemical industries, will be received as valuable to many. Experience gained in any branch of industry is not only valuable to those engaged in this branch, but the same may be many times most usefully transferred to new channels. Communications of this nature are therefore often more valuable to the technical chemist than the mere fact of the discovery of some complicated organic compound, which may probably never enter into the precincts of our works, however important the same may be to the scientist. We ought to draw a distinct line of demarcation between industrial and purely scientific chemistry, and concentrate ourselves on such subjects which only come within our province, and not import matters which are neither intelligible reading to the bulk of our members nor in any way connected with manufactures.



MANCHESTER TECHNICAL SCHOOL, *Tuesday, Feb. 5th.*

MR. IVAN LEVINSTEIN IN THE CHAIR.

## THE SEPARATION OF BENZENE, TOLUENE, AND XYLENE, FROM CRUDE GAS-TAR NAPHTHA AND CRUDE GAS BENZENES.

BY J. VON HOHENHAUSEN.

A NUMBER of valuable papers have lately been read before this Society on the carbonisation of coal with the object of increasing the quantity either of ammonia or of benzene and its homologues, toluene and xylene. I thought, therefore, that it might not be out of place to make a few observations on the processes and apparatus employed in the separation and production of these substances.

Up to quite recently these commercially important hydrocarbons were only produced from gas-tar naphtha, but the carbonisation of coal with the view of obtaining benzene from gas has furnished a new source of supply. The benzene extracted by passing the gas through fatty absorbents and then recovering

it from the latter by distillation, is not pure benzene, but is accompanied, in addition to toluene and xylene, by other compounds, from which the three former may be separated by fractional distillation. The separation can be effected in a similar manner to that adopted in the production of these hydrocarbons from crude gas-tar naphtha. It will therefore suffice to describe the methods and apparatus used for obtaining these hydrocarbons from the gas-tar naphtha.

Crude naphtha, or "first runnings," is a well-known

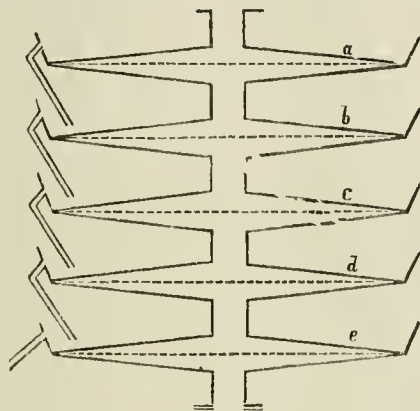


Fig. 1.

term in the trade, which is applied to the portion first coming over in the distillation of tar. It is generally sold and its value fixed according to its distilling points. The quality of naphtha varies considerably. The following are the distilling points of some fair average naphthas from different places, of which I have used up considerable quantities:—

Centigrade.	Wigan. Per cent.	Yorkshire. Per cent.	Scotland. Per cent.
105 .....	—	1	—
110 .....	4	16	17
120 .....	19	34	38
130 .....	33	47	49
140 .....	45	—	58
150 .....	55	—	69

I have also had an opportunity of rectifying French crude naphthas from the Mines d'Anzin and the Mines de Blanz, in France, and from the Compagnie Parisienne de Gaz. The following are the results:—

Centigrade.	D'Anzin. Per cent.	Blanz. Per cent.	Comp. Paris. de Gaz. Per cent.
100 .....	5	14	10
120 .....	24	37	23
130 .....	39	49	35
140 .....	50	59	47
150 .....	60	69	58
160 .....	67	77	69
170 .....	75	85	—

Whatever may be the quality of the naphtha, it ought

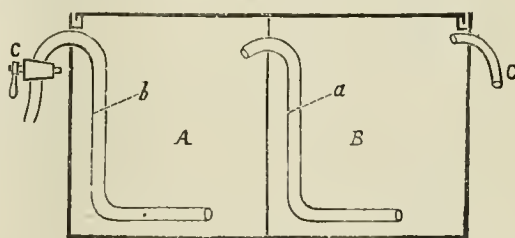


Fig. 2.

always to be purified before proceeding to rectify or separate it. The purification consists of washing the naphtha first with caustic soda lye, and afterwards with concentrated sulphuric acid. The quantity of vitriol used in washing depends to some extent on the quality of the crude naphtha, but generally one



pound of vitriol suffices for one gallon of naphtha. A great point is the thorough mixing of the naphtha with the acid, otherwise the solvent naphtha afterwards obtained in the rectification will be apt to turn rather dark after being kept for some time. The naphtha thoroughly washed is then submitted to separation in a dephlegmator. The construction of this apparatus, as used at the beginning of this industry, and still in use in some refineries, is illustrated in Fig. 1. It consists of a wrought-iron still, provided with 5 copper pans *a, b, c, d, e*, of one metre in diameter. Each of these is divided internally by a perforated copper plate. The vapours of the lower boiling naphtha passing through the perforated plates partially condense those of the higher boiling, which thus condensed flow back into the still. Water enters into the outside of pan *a*, and flows over into *b*, and so on until it passes out from *e*. The still is connected with a copper or iron worm, and can be heated by wet or dry steam. When charged, care must be taken to insure continuous and regular distillation. On starting the distillation, dry steam is turned on, and when the distillation begins to slacken—that is, after nearly all the benzene has come over—wet steam is turned into the still, and the distillation continued so long as any hydrocarbons come over. The water may be separated from the naphtha by an arrangement shown in Fig. 2. This is a cistern made of tinplate, which is divided into two compartments, A and B, which are connected by the pipe *a*. The benzene and water run into A. The benzene being lighter then passes through the pipe *a* into B, overflowing at G into receiving cisterns. The water discharges through pipe *b*, the latter being provided with a stop-tap at *c*. If the distillation and separation in the dephlegmator are properly carried out the results will be as follows—Charge: 500 gallons of Yorkshire crude naphtha, which tested before separation

%	C.
16	at 110
31	120
47	130

The first portion, collected from the dephlegmator, by distillation with dry steam, yielded about 250 gallons of the following distilling points:—

%	C.
15	at 95
39	100
75	110
89	120

Such distilling naphtha is called in the trade a 40 per cent. benzene. Several qualities of Wigan crude naphtha gave under the same circumstances the following results:—

SAMPLE A.		First portion collected.	
500 gals. tested.	% C.	Yield 200 gals.	C.
17	at 110	—	—
38	120	11	at 95
49	130	38	100
58	140	73	110
69	150	89	120

SAMPLE B.		First portion collected from the still.	
500 gals. tested.	% C.	Yield 200 gals.	C.
13	at 110	—	—
31	120	15	at 95
47	130	40	100
57	140	71	110
68	150	90	120

From these examples it will be seen that a fair average English crude naphtha, yielding about 35 per cent. at 120° C., will give 50 per cent. or thereabouts of commercial 40 per cent. benzene. The second portion collected from the distillation, which contains non-nitrifiable hydrocarbons mixed with some toluene, xylene, and cumene, was generally sold

as solvent naphtha, or was again subjected to fractional distillation, in order to recover some toluene and crude xylene.

A dephlegmator of improved construction is shown in Fig. 4. It is made by Egrot, of Paris, and we will shortly refer to it again. By this apparatus 30, 50, and 90 per cent. is produced in one distillation. Before proceeding to the description of the different systems used for the separation and production of pure benzene, etc., it may be of interest to mention a few practical tests of commercial benzene. The principal points to be taken into account are, (1) specific gravity; (2) distilling points; (3) treatment with sulphuric acid; (4) nitric acid of the benzene. The specific gravity of a good commercial 30 per cent. English benzene is generally 0.875 at 15° C., or 60° F.; of a 50 per cent., 0.878; and of a 90 per cent., 0.882. When the specific gravity is lower than 0.875 the presence of paraffin or non-nitrifiable hydrocarbons may be suspected. Most of the Scotch benzene possesses a lower specific gravity than this. The average Scotch benzene has a specific gravity of 0.870, and often contains 7 to 8 per cent. of paraffins. Such benzenes give a smaller yield of their respective nitro and amido compounds; and 100 parts of a fair average English 90 per cent. benzene yield about 150 parts by

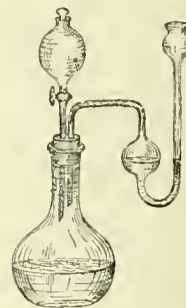


Fig. 3.

weight of well-washed nitrobenzene, whilst 100 parts of some 90 per cent. Scotch benzenes do not yield more than 135 per cent. nitrobenzene. A good average commercial 30, 50, and 90 per cent. benzene ought to distil within about the following points:—

90 Benzene.	50% Benzene.	30% Benzene.
Spec. Gr. 0.882.	Spec. Gr. 0.878.	Spec Gr. 0.875.
% C.	% C.	% C.
5 at 83	10 at 91	12 at 97
22 85	18 95	21 98
62 88	40 98	30 100
74 90	50 100	55 105
81 92	68 105	73 110
87 95	79 110	84 115
90 100	85 115	90 120
94 105	85 115	
98 110	90 120	

A well-washed and rectified benzene gives with sulphuric acid a dark yellow or light brown colour. A dark brown or even black colour indicates bad washing and defective purification. The sulphuric acid used in these tests always looks rather darker when mixed with 30 and 50 per cent. than with 90 per cent. benzene.

The nitric acid test of commercial benzene may be easily performed by putting 100 c.c. in a flask of 500 c.c. capacity (see Fig. 3), and gradually adding to it a cold mixture of 150 grms. nitric acid, 80 Tw; and 200 grms. sulphuric acid, 170° Tw. When the mixture of the benzene with the acids becomes warm, cool it; then add some more of the acid until all is used up; mix well all the time by shaking the bottle, and continue this, after the whole of the acid has been added, until the contents become cold; then separate the acid from the nitrobenzene which has been

formed, wash the latter several times with a dilute solution of caustic soda, and afterwards with water; let the liquid settle for about 12 hours, and then separate the nitro compound. This test is not only important because of its determining the yield of nitrobenzene, but it also gives a fair idea of how the benzene is likely to work on the larger scale. A good quality of benzene should not get hot immediately on a small portion of the acid being added to it at first. If 100 c.c. of nitrobenzene made in the above manner are put in a retort and about 98 per cent. distilled over, the 2 per cent. residue must remain liquid.

A further test of the quality of the benzene to be examined is to distil the whole of the nitrobenzene obtained from 100 c.c. of benzol, and to treat the first 30 c.c. which distil over with 70 c.c. concentrated sulphuric acid, 170° Tw., when the nitrobenzene will dissolve in the vitriol, and all the non-nitrified hydrocarbons will separate. These hydrocarbons are again treated with a small quantity of nitro-sulphuric acid mixture, and in case benzene is present there will be a rise of temperature. The waste acid is then drawn off, and the untransformed hydrocarbons, if any be present, mixed with sulphuric acid, the quantity of hydrocarbon which is not soluble in the sulphuric acid giving the percentage of paraffins present. The specific gravity of nitrobenzene made from 90 per cent. benzene is by Baumé's hydrometer 25°; 50 per cent., 23°; and 30 per cent., 22°. The coal-tar colour manufacture in its present state of development requires aromatic hydrocarbons of great purity. It therefore becomes of the highest importance to employ the most perfect appliances for their proper separation. I thought, therefore, that it might be of interest to lay before you this evening a description of the apparatus best calculated to attain this object. You will notice that the apparatus consists of four parts, (1) the steam still; (2) the rectifying column; (3) the condenser or *analyseur*, as the French call it; (4) the cooler. The differences in the various systems principally used by rectifiers of benzene may be summed up as follows: Savalle, of Paris, passes the naphtha vapours direct into the column, and his condensers, made of copper, are furnished with copper tubes, through which cold air is forced, whilst the naphtha vapours pass round the pipes thus cooled. Egrot (see Fig. 4) condenses the vapours by passing them through a large copper coil fixed in an iron cistern, and through which water passes. Vedles (see Fig. 5) passes the vapours through four copper cylinders, which are surrounded by water. The vapours pass from the rectifying columns—first into one of the upper cylinders, then through a large pipe into the cylinder immediately below it, and from thence they pass into the second under cylinders, and from thence into the second top cylinder, and finally into a condenser of the shape of the letter U.

#### J. ADERS' APPARATUS.

Unfortunately I am not able to give you an illustration of this rectifier. I wrote to Aders for a sketch showing the construction of his apparatus, but received the reply that he only supplies a drawing after an order has been given, and as the cost of the apparatus is about £1,000, the expense would be rather too great to incur for the sake of this evening's paper. As far as I can gather from information supplied, Aders does not pass the vapours direct from the still into the column, but conveys them sideways into the rectifier, which I consider an improvement, as the vapours thus do not come into contact with the condensed higher boiling hydrocarbons, which run back from the rectifying column into the still. Aders considers his arrangement of steam coils for heating the

still, consisting of 3in. copper coils with a large number of joints, about 25, as an important feature. His steam-pressure regulator is similar to Savalle's (Fig. 7). In the absence of fuller information, I should not incline to think it an advantage to work with such a number of joints, which are very liable to leakage, especially towards the end of the operation, when the steam pressure is highest. Instead of having coils for heating the naphtha inside the still, I prefer to use a still surrounded by an iron mantle, or a jacketed still as it is called. In such a still the steam can be easily regulated, and so long as it is provided with safety valve, etc., there is no danger. On the other hand, leakages are very serious in the case of internal coils; and there is the danger of loss of material before the leakage is found out, and when the fact is ascertained the whole apparatus must be taken to pieces, which involves loss of time, etc. The condenser of Aders consists of copper tubes similar to those of Savalle, with this difference, that they are supplied with water instead of air. With the limited information which I at present possess I cannot state whether Aders represents his apparatus as an absolutely new construction, but I used a very similarly-constructed rectifier more than five years ago for making pure toluene at Vedles' works, in Paris, of which I will presently give you a description. Nevertheless, it is not my intention to disparage Aders' apparatus. It is in use on the Continent, and, if I am correctly informed, an English aniline company, which has already had one from him, has recently ordered a second one. It is also in use in another works in this country.

In considering the comparative merits of the different systems from the practical point of view, I must confess that I do not think there is any very material difference, either in the results obtained or in the cost of production of the different hydrocarbons; for, after all, Aders does not pretend to obtain more than 70 per cent. of pure benzene from 90 per cent. benzene, and a similar quantity is got with Savalle's and Vedles' apparatus. If it is a question of economy in the erection of plant, and it is intended to make chiefly commercial 30 per cent., 50 per cent., and 90 per cent. benzene, I would recommend Egrot's rectifier, the cost of which is not half that of any of the above-mentioned apparatus.

We will now go more into the details of the construction of some of the so-called column rectifiers, beginning with that of Vedlè.

#### VEDLES' TOLUENE APPARATUS.

This apparatus consists of a wrought-iron double-cased still A (Fig. 6), provided also with a perforated copper coil for heating the contents, when required, by means of open steam. The still is 1,800mm. in diameter and 1,800mm. deep. The still is surmounted by the dephlegmating column B, which is of similar construction to that shown as the benzene dephlegmator. The vapours leaving the dephlegmator pass out by the pipe C and enter the condenser or analyser D, which consists of a cylindrical or square copper cistern, 1,500mm. deep by 600mm. diameter, in which is fixed a number of copper tubes, 1 metre in length and 30mm. diameter. There are about 60 tubes in this analyser, and through them the condensing water is made to pass, whilst the hydrocarbon vapours fill the space around the tubes. The uncondensed vapours pass out by means of the pipe E and enter the cooler F, of similar construction to the analyser D. The higher boiling hydrocarbons, which have been condensed in the analyser, run back through the pipe *a* into the rectifying column. The distillate runs from the cooler F through the pipe H into the receiving cisterns. The condensing water enters the



cooler through the tap J, and is fed to the analyser by means of the tap K. The waste-water pipes may be easily recognised.

When the still A is charged with the residues from the manufacture of pure benzene it is first heated by means of the steam jacket, and finally wet steam is slowly admitted, the progress of the distillation being carefully regulated. When the distillate begins to run slowly, the water supply is slackened by means of the tap K until the water in the analyser D commences to boil. The distillate is collected in separate cisterns, or better in 100 gallon drums, which are numbered and separately analysed. When the distillate begins to run slowly from the cooler F, while the water in the analyser is boiling, the tap  $g^1$  is opened. The overflow of the water now runs away through the pipe connected with the tap  $g$ , the space in the analyser above this being filled with steam. In regulating the taps  $g^2$ ,  $g^3$ ,  $g^4$ , each time the distillation begins to flag a mixture of toluene and xylene is distilling. At the end of the operation the tap K is completely shut and the analyser becomes filled with steam. The distillation of xylene then commences and the operation is concluded when no more comes over at  $142^\circ \text{C}$ . By analysing the contents of the drums the rectifier finds at once which contain pure toluene and pure xylene and which contain intermediate products.

#### EGROT'S SYSTEM.

In this system the dephlegmating column A (Fig. 4) is made of copper 530mm. diameter, the interior being fitted with 22 partitions. C is a copper pipe conducting the vapours from the column to the condenser. The condenser D consists of a large copper coil lying in a wrought-iron tank. E is a tap and pipe connecting the cistern D with the column, and through which water is passed for cleaning purposes. F F F are small taps, which connect the condenser with the tube G. I I I are three-way taps. The higher boiling hydrocarbon vapours, after condensation, run through the tubes F F F into the tube G, and from thence into the column A through the tubes H H H or through the tap J into the cooler L. The three-way taps permit the condensed higher boiling hydrocarbons to run back into the column, but when 90 per cent., 50 per cent., or 30 per cent. benzenes are required the liquids are allowed to run through the pipe K into the cooler L. These taps also permit a portion of the liquid to be run each way. L is a wrought-iron cistern containing the cooler. M is the tube connecting the cooler with the glass test apparatus N, through which the condensed hydrocarbons are run into the receiving cisterns. O is the tap for passing cold water into the cooler, the waste water passing away through the tube Q into the top of the cistern D. The tap for discharging the cooler cistern L is shown at P.

Having had some experience of this rectifier I can testify to its working very steadily. The yield of pure benzene from 90 per cent. benzene is slightly inferior to Savalle's apparatus, and also to that of Vedles. Egrot's rectifier yields 650 gallons from 1,000 gallons charged, and is an admirable apparatus for making commercial benzenes from crude naphtha.

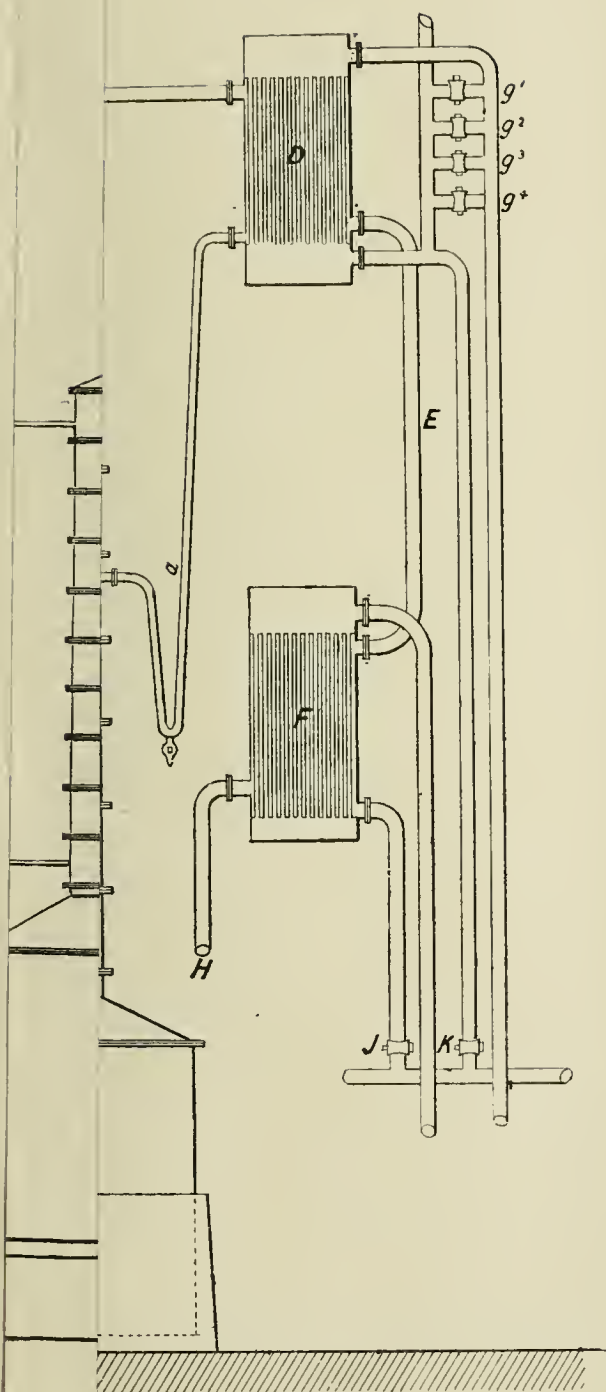
#### VEDLES' SYSTEM.

This apparatus, as sketched, will turn out 8,000 gallons of pure benzene monthly. The still A (Fig. 5) is of wrought iron, 1,800mm. in diameter and 1,800mm. deep, in the inside of which is a copper coil for close steam. Later on I changed this still for one heated by means of a steam jacket, as with the coil there was much trouble, with constant leakages. The still is in connection with the usual dephlegmating column, com-

posed of ten compartments of cast iron 600mm. in diameter. These compartments contain a number of partitions inside them, arranged so that the vapours pass through condensed liquid on each partition. The vapours leave the dephlegmator, and pass by means of a copper pipe into the condenser, which consists of four copper cylinders lying in a square wrought-iron tank. Each of the cylinders is 2,300mm. in length and 400mm. in diameter. The vapours pass into the upper cylinder on the right hand, and descend into that one immediately below it, then to the lower one on the left hand, and after rising into the top left-hand cylinder pass away from this into the U tube, from which the uncondensed vapours pass into the cooler. The vapours which are condensed from the copper cylinders, and also from the U tube, pass back by means of the pipes into the dephlegmator column. The cooler is not a copper coil, but consists of a copper cylinder 2,900mm. long and 400mm. wide, and fixed in a wrought-iron cistern. The water enters at the bottom and runs through the cistern, passing out at the top, and enters the bottom of the tank containing the U tube, and leaving this tank supplies the first condenser containing the copper cylinders with water. It is of great importance that no direct or wet steam be used in the preparation of pure benzene, as the water would condense in the small back-flow tubes, and form a pressure which would prevent regular distillation.

The following table extracted from my diary gives an account of the working of a benzene rectifier (Vedles). The still is charged with 1,000 gallons of 90 per cent. benzene. The distillate is collected in iron drums capable of holding about 100 gallons each. The two first drums contain about 160 gallons distilled under  $81^\circ$ . This portion is put aside until there is a sufficient quantity, say about 1,000 gallons, when it is again separated by distillation. All that comes over between  $81^\circ$  to  $82^\circ$  is commercially pure benzene. The contents of Drum IX. are redistilled with a fresh lot of 90 per cent. benzene. The residues remaining in the still after the fractional distillation has been stopped are transferred to the toluene apparatus (Fig. 6), where pure toluene is separated from them. 1,000 gallons of 90 per cent. benzene, after complete separation, yield about 80 gallons of compounds, distilling  $40^\circ$  to  $80^\circ \text{C}$ .; 710 to 720 gallons of commercial pure benzene,  $81^\circ$  to  $82^\circ \text{C}$ .; 165 gallons of residues or crude toluene,  $100^\circ$  to  $130^\circ \text{C}$ .; 35 to 45 gallons of loss, volatilised during manipulation.

With regard to Egrot's rectifier, which is a very convenient apparatus for transforming well-washed crude naphtha into 30, 50, or 90 per cent. benzene, I wish to add that the hydrocarbons which are condensed during the process of distillation may be conveniently collected in three covered iron cisterns. The first portion of the distillate is run into cistern I, the next into cistern II., the third and last into cistern III. During the distillation samples are drawn and tested, and in this way there is no difficulty in collecting in cistern I. 90 per cent. benzene, in No. II. toluene, and in No. III. solvent naphtha. By then mixing the hydrocarbons from cisterns I and II. in certain proportions you get the corresponding qualities of commercial 30, 40, 50, or 70 per cent. benzene. The solvent naphtha may be redistilled in the same apparatus for the purpose of separating from it crude xylene. The very best arrangements of apparatus for rectifying do not, however, produce chemically pure but only commercially pure benzene. Chemically pure benzene is obtained by crystallising commercially pure benzene in a freezing mixture. The benzene thus crystallised is known as chemically pure benzene, or as benzene purissimum crystallisatum. Such benzene is a colourless liquid, possessing a specific gravity of



S' TOLUENE RECTIFIER,—FIG. 6.

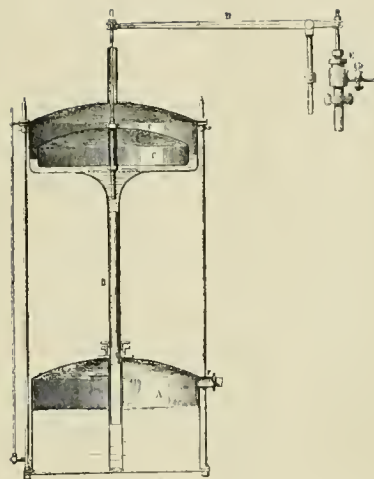
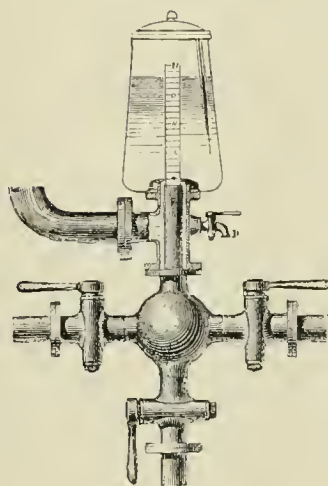
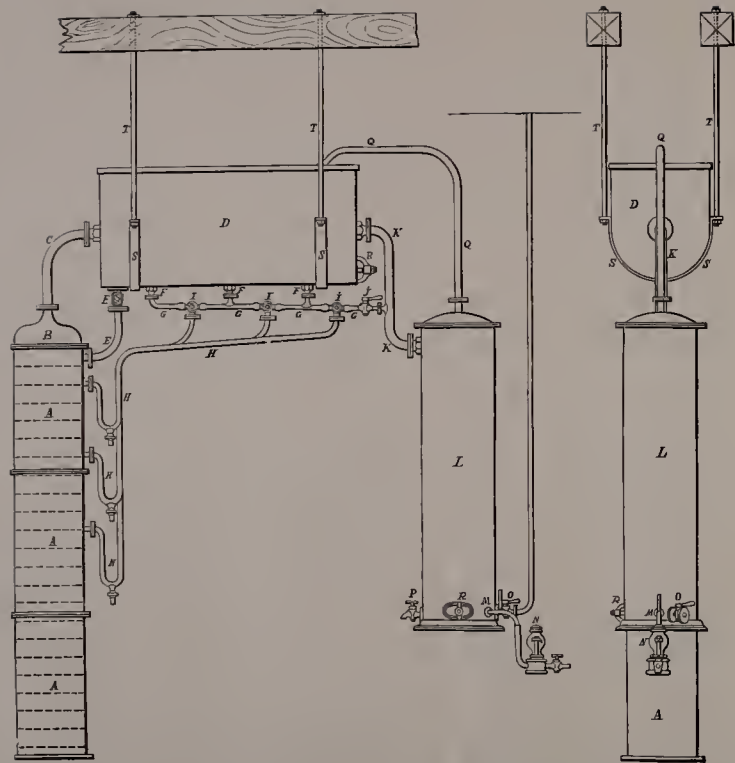


FIG. 7.

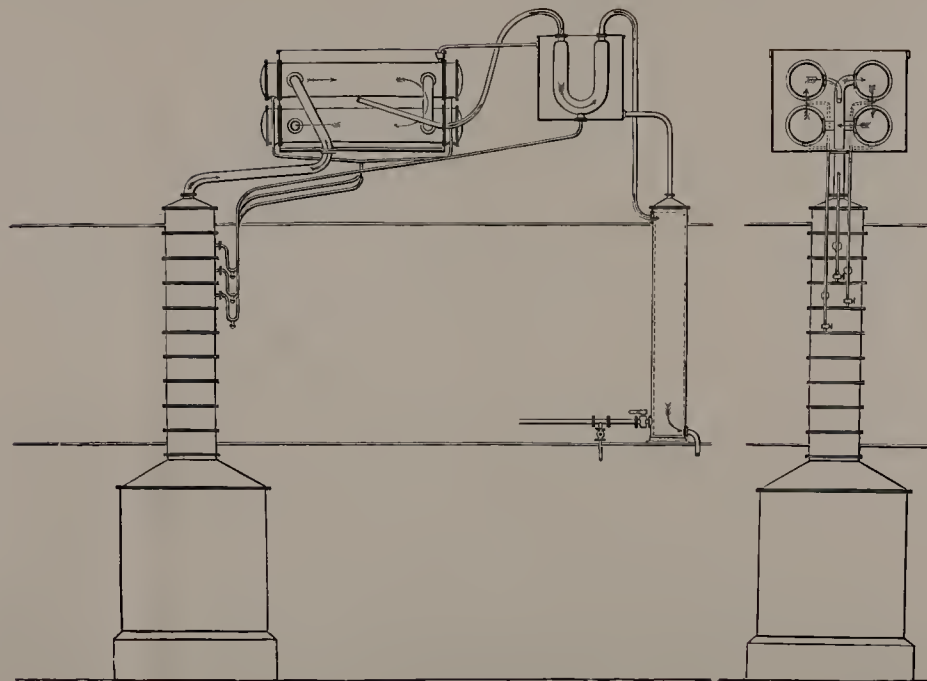


SAVALLE'S TEST APPARATUS.

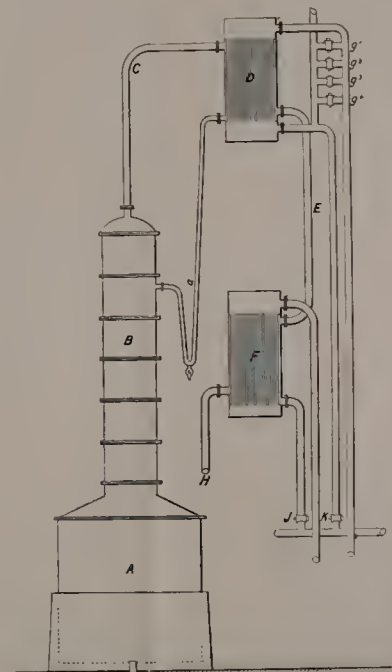




FROST'S RECTIFIER.—FIG. 4.



VEDER'S BENZENE RECTIFIER.—FIG. 5.



VEDER'S TOLUENE RECTIFIER.—FIG. 6.



FIG. 7.



FIG. 8.

0.899 at 0° C., and boiling at 80.5°. Until about eight months ago benzene purissimum crystallisatum, as the name clearly indicates, was considered an absolutely pure product, but about that time the eminent chemist Professor Victor Meyer showed, after a most cleverly-conducted investigation, that even such benzene is not pure, but contains about 0.5 per cent. of a compound hitherto unknown to chemists, to which he gave the name of thiophene. Its chemical composition is  $C_4H_4S$ . It is interesting to note that Mr. George E. Davis, in a paper read in December last, before the Birmingham Section, states that by crystallising gas benzene he separated a compound which, in his opinion, is probably thiophene. Chemically pure toluene is also a colourless liquid, with an odour similar to that of benzene. Its specific gravity is 0.882, at 0° C.; boiling point 111°.

There is no chemically pure xylene produced for commercial purposes. That which is manufactured is sold at a price which precludes its use in commerce. Manufacturers of xylidine buy crude xylene. Such a product distils within two or four

The CHAIRMAN: I think great credit is due to Mr. Hohenhausen for the trouble which he has taken in bringing before the Society in such a clear and elaborate manner the different apparatus used in the separation of the three important aromatic hydrocarbons—benzene, toluene, and xylene; and I also think the information which he has given us must be welcome to all refiners who have not got the most improved appliances desirable to obtain this object. In the present high development of the chemical industries it becomes a matter of necessity to employ the most improved apparatus, otherwise manufacturers will be at a disadvantage in producing pure articles in an economical manner. At present the Germans are the largest buyers of the raw materials referred to, and one would think that it must pay our refiners much better, considering the high freight, etc., to export these products in as pure a state as possible. We have to thank Mr. Davis for the admirable manner in which Mr. Hohenhausen's drawings have been illustrated by means of the lantern. With your permission, gentlemen, we will postpone the dis-

## BENZENE APPARATUS.

## TOLUENE APPARATUS.

Charge.	No. of collecting drums.	Quantity in gallons.	Degrees of distillation of the different fractions collected in the drums.							Remarks.	Charge.	Quantity.	Remarks.
10 puncheons marked O.V.	I.	100	70°	75°	79°	80°	81°	82°	83°	Drums I. and II. Quantity 160 gals., kept for redistillation. Drum IX. went with next lot of 90% benzene. Quantity 60 gals. The residues in the still, quantity 165 gallons, were transferred to toluene apparatus.	Residues from benzene still.	165 gallons	This apparatus was only charged when a sufficient quantity of residues had accumulated.
130/139, and containing 1000 gals. of 90% benzene.	II.	60	2%	40%	65%	80%	92%	98%	—		Spec. grav. 0.872. Degrees of distillation.	5% at 100° C.	
0.882, distilling 20% at 85° C.	III.	100	—	—	—	20%	90%	98%	—		30% 105° C.	55% 110° C.	
76% 90° C.	IV.	100	—	—	—	—	1%	98%	—		72% 115° C.	87% 120° C.	
86% 95° C.	V.	100	—	—	—	—	1%	98%	—				
91% 100° C.	VI.	100	—	—	—	—	1%	98%	—				
91% 105° C.	VII.	100	—	—	—	—	1%	98%	—				
98% 110° C.	VIII.	100	—	—	—	—	1%	98%	—				
	IX.	60	—	—	—	—	—	50%	90%				

or five or more degrees. Its composition, however, varies, as Mr. Levinstein, who has had perhaps more experience in the separation of pure xylene than any other chemist in this country, will no doubt demonstrate to our satisfaction this evening.

Before finishing this paper I desire to refer to the intermediate products distilling from 40° to 80° C., from 82° to 110° C., and from 112° to 135° C. Rectifiers of aromatic hydrocarbons are dealing to-day with different products from those of 10 or 15 years ago. At that time there was little demand for pure benzene or pure toluene. The commercial benzenes were mostly only the first portion of purified crude naphtha, from which no pure benzene or toluene was separated. Since that time there has been a large demand for pure benzene, pure toluene, respectively for pure aniline and pure toluidine. The question then arises, what is done with these intermediate products, some of which are neither benzene nor toluene, while others contain benzene and toluene mixed with other hydrocarbons or compounds different from benzene, toluene, or xylene. As a matter of fact, there are refiners—let us hope they are only a small minority—who use these intermediate products for the purpose of sophisticating commercial benzenes and toluenes. I cannot, therefore, lay too much stress on the necessity of carefully testing the commercial benzene, and not relying, as is often done, only on the degrees of distillation, and I trust that the few practical tests mentioned will be helpful in detecting such adulterations.

cussion on Mr. Hohenhausen's paper while I read to you a short communication

## ON THE QUANTITATIVE DETERMINATION OF ORTHO-, META-, AND PARA- XYLENE.

BY IVAN LEVINSTEIN.

XYLENE is a hydrocarbon belonging to the aromatic series, but different from benzene. It is known to exist in three modifications, viz.:—

Orthoxylene boiling at.....	111–112° C.
Metaxylene boiling at.....	140–141° C.
Paraxylene boiling at.....	136–137° C.

It is, however, only since 1877 that all these three modifications have been known to be generally present in gas-tar naphtha distilling at from about 134° to 140°. Indeed, in the latest edition of Miller's Organic Chemistry, date 1880, revised by Armstrong and Groves, we read at page 285: "Xylene, from coal tar: The portion boiling at 136° to 140° contains para- and meta- xylene in variable proportions, but usually consists mainly of metaxylene. Other hydrocarbons, however, are present, the nature of which is unknown." This shows that the revisers of this otherwise very useful book were, at the time of writing the paragraph in question, unaware of the existence of orthoxylene in such naphthas, although in the additions, corrections, and cross-references at the end of the book there is a reference on page 1003 to Jacobsen (Ber. 10, 1009). Until a few years ago xylene in an isolated form was not a commercial article, but was met with in variable quantities mixed with other hydrocarbons in solvent



naphtha, a naphtha largely used by manufacturers of indiarubber goods. Suddenly a demand sprang up for xylene pure and simple as one of the starting points for the manufacture of naphthol-xylidinescarlet, and as frequently happens with new products, there was at first some difficulty in procuring it. This difficulty has now been overcome. There is still, however, another remaining, and that is the want of a ready method of determining its commercial value. Xylene is principally produced by fractional distillation of solvent naphtha, or of the higher boiling portion of crude naphtha, and with proper appliances, such as have been shown this evening by Mr. von Hohenhausen, there is no difficulty in obtaining it to distil within two degrees. You might then think that a hydrocarbon boiling at its proper degree would be pure enough for all commercial purposes, and no doubt such is the case with benzene and toluene. It is, however, different with xylene. As I have already stated, there are three modifications of it known, of which the meta is the only one possessing technical value, whilst as far as I am aware there exists at this moment no practical use for the ortho- and paraxylenes. Considering then that the distilling degrees of these modifications are so close to each other, and that their relative proportions vary in commercial xylene, you will at once comprehend that it is impossible to separate the different xylenes from each other by fractional distillation, and that the distilling degrees are altogether misleading, so far at least as the composition of commercial xylene is concerned. Notwithstanding this, contracts between buyers and sellers of this article are still being made, probably for want of a better basis of description, by specifying simply the degree of distillation. The wording is generally: "(So many) gallons of xylene of well-washed quality, distilling within (such and such) degrees." The consequence is that buyers with no other specification of the quality of xylene will sometimes find out to their sorrow that they have made a very foolish bargain. I have endeavoured to devise some practical methods for the quantitative determination especially of the metaxylene, which I now have the pleasure of submitting to you.

#### (A) METHOD FOR DETERMINATION OF METAXYLENE.

Take 100c.c. of the crude xylene, heat them at 100° C. in a suitable glass vessel on a sand-bath, together with dilute nitric acid (40 c.c. acid 80° Tw. and 60 c.c. of water) from half an hour to an hour, or until red fumes no longer are evolved, taking care that the acid is thoroughly mixed with the hydrocarbon; then separate the latter from the acid, and add to the mixture of hydrocarbons an excess of caustic soda, and distil the whole by wet steam. The distillate consists of water and metaxylene, mixed with paraffins, which are always present in commercial xylenes. The hydrocarbons are then separated from the water and about  $1\frac{1}{2}$  of their own volume of concentrated sulphuric acid is added and well mixed for about half an hour, when the metaxylene will be dissolved, whilst the paraffins remain unaltered. Instead of distilling the mixture of hydrocarbons and caustic soda, several times washing with the latter will be also sufficient. By deducting the number of cubic centimetres of paraffins from the original measure in cubic centimetres of the mixture you will get the percentage of metaxylene in the crude sample. Dilute nitric acid converts the ortho- and para-xylene into toluic acids, or into their respective nitro-compounds. These are soluble in caustic soda, and remain behind in the distillation, whilst the metaxylene, under the above-mentioned circumstances, is very little altered by the acid, and distils with the water and the paraffins.

#### (B) METHOD FOR DETERMINATION OF PARAXYLENE.

Take 100c.c. of the crude xylene; add to it about 120 c.c. concentrated sulphuric acid (170° Tw.); mix the whole for half an hour or so, or until no longer any crude xylene is dissolved, and allow it to settle. It is advisable to cool the mixture. The hydrocarbons not acted upon, consisting of paraxylene and paraffins, will rise to the top; separate them, and add to them equal volumes of fuming sulphuric acid containing 20 per cent. of anhydride; heat on a water-bath for a short time; then allow the liquid to settle. The paraffins will rise to the top. Deduct the quantity in cubic centimetres of the latter from the quantity of the original mixture of paraxylene and paraffins, and you will get the percentage of paraxylene present in the sample of crude xylene. Ortho- and meta-xylene are converted into sulphonic acids by the action of ordinary concentrated sulphuric acid, whilst the paraxylene is not altered. Fuming sulphuric acid likewise converts the para modification into a sulphonic acid, but does not act on the paraffins. When it is desired to separate the paraxylene from the paraffins without converting the former into a sulphonic acid, and at the same time to obtain absolutely pure paraxylene, I found the following method to give good results: Put a mixture of the two into a glass flask connected with a Liebig condenser, and convey wet steam into it. The first portion distilling along with the condensed steam is principally paraxylene. Separate this from the water, and distil it once more by applying external heat. Collect all that comes over up to 138° C., and put this fraction into a freezing mixture, when paraxylene crystallises out, and in order to obtain it absolutely pure press the crystals between filter paper.

#### (C) METHOD FOR DETERMINATION OF ORTHOXYLENE.

If the crude gas-tar xylene represents a fair average commercial quality, and originates from English tar, the quantity of orthoxylene present in the sample to be examined may be estimated by calculation. Add together the amount of metaxylene, paraxylene, and paraffins determined by methods A and B; the difference between the whole and 100 will give the quantity of orthoxylene. For example, if 100 c.c. of crude xylene were found to contain 47 per cent. of metaxylene, 8 per cent. of paraxylene, 26 per cent. of paraffins, then the quantity of orthoxylene would be 19 per cent.; total, 100. But as dilute nitric acid also alters the composition of other aromatic hydrocarbons than ortho- and para-xylene, such as toluene, cumene, and cymene, and also acts on other compounds which may possibly be present in crude xylene, it will be obvious that such an estimate by calculation can only be approximately correct. Nevertheless, I generally found this method to give fairly good results with unsophisticated crude xylene properly separated from English gas-tar naphtha, as in such xylene there is, as a rule, no toluene present, and only small quantities of compounds acted on by dilute nitric acid. Should it, however, be of importance to ascertain whether the difference, as calculated by method C, really consists mainly of orthoxylene, and whether the quantity thus determined is approximately correct, it will be advisable to proceed as follows:—

Employ part of method B to effect the separation of the paraffins and the paraxylene from the crude xylene; convert the meta- and ortho-xylene sulphonic acids into their calcium and afterwards into their sodium salts; concentrate the solution of the latter, when the ortho-salt will crystallise in large prisms,

while the meta-salt remains in solution; concentrate the solutions still more, and recrystallise the first and second crop of crystals. The ortho-salt will then be obtained in large well-developed crystals. If you concentrate the solution too far, some of the meta-salt will be mixed with it, which, however, will be nearly all removed by recrystallisation. There is no difficulty in distinguishing the large well-defined crystals of the ortho-salt from the indistinct crystals of the meta-salt. I have, however, observed that the complete separation by crystallisation of the sodium salt of the orthoxylylene sulphonic acid from the sodium salt of the metaxylylene sulphonic acid is much more difficult with Scotch than with English xylenes. Some Scotch samples, containing as much as 10 per cent. of orthoxylylene, did not produce as well-developed crystals, even after several recrystallisations, as some English samples gave after only one crystallisation, notwithstanding that they did not contain perhaps more than 3 per cent. of orthoxylylene. The sodium salt of the orthoxylylene sulphonic acid crystallises with five molecules of water. Its formula is—



The difference in the crystallisation of the sodium salt of the orthoxylylene sulphonic acid made from Scotch xylene is very remarkable, and I cannot at present account for it, otherwise than that the Scotch xylene probably contains some non-saturated hydrocarbons or other compounds which dissolve in the sulphuric acid and form sulphates and sulphonic acids, and thus interfere with the crystallisation of the ortho-salt. I have only made a few preliminary tests, but I noticed that the Scotch xylene absorbed much more rapidly aqueous solutions of bromine than the English xylene. In order to establish the true cause of this difference between Scotch and English xylene it would be desirable to investigate the oxidised products obtained by method A and the sodium salts\* of the sulphonic acids obtained by method B. Unfortunately, however, time does not allow me to continue these experiments. I have mentioned this matter to Mr. Watson Smith, of the Owens College, and I am glad to say that he has kindly consented to further investigate it. For all practical purposes, however, method A will be quite sufficient, as it is at present of very little importance to the consumer of xylene how much ortho- or how much para- xylene, or how much of other compounds oxidisable by dilute nitric acid the sample contains. All that he desires to know is the quantity of metaxylylene present in the crude xylene which he intends to purchase; but as method B gives very little trouble I would recommend its use as a means of checking the results of method A.

The table of analyses given below represents about a dozen samples of fair commercial xylenes. The determinations have been made according to methods A, B and C; the presence and approximate quantity of the orthoxylylene has in each case been established by separating and recrystallising the sodium salt of its sulphonic acid. In every instance crystals of the ortho-salt were obtained and further verified as such by analysis. These determinations show that the distilling degrees do not in any way indicate the good quality of xylene. (Compare samples B and L.) Sample L was sold as commercially pure xylene, and no doubt it distilled within 2 degrees; nevertheless it did not contain more metaxylylene than sample I, which was sold at almost half the price, etc. Crude xylene of the same distilling degree and made from English

tar generally contains more metaxylylene than Scotch xylene (compare samples B and F), whilst Scotch xylene and mixtures of Scotch and English are richer of paraxylylene and paraffins, and their specific gravity is lower than that of English xylene.

Sample.	Origin.	19° C. Spec. Grav.	Dist. deg.	Metaxylylene. %	Paraxylylene. %	Orthoxylylene. %	Paraffins. %
A	Pure meta obtained by method A from English crude xylene.....	0·8668	142-113	100	0	0	0
B	English (Manchester tar)	0·8629	134-110	87	6	4	3
C	English .....	—	110-143	87	4	6	3
D	English .....	—	141-145	83	5	7	5
E	English .....	0·8660	138-111	79	3	15	3
F	Scotch .....	0·8571	134-110	72	8	12	8
G	Unknown .....	—	139-111	76	5	15	10
H	English and Scotch mix'd	0·8605	134-141	81	10	3	6
I	English and Scotch mix'd	—	136-112	86	6	1	1
K	English and Scotch mix'd	0·8613	136-111	86	6	2	6
L	English and Scotch mix'd	—	110-141	86	3	5	6
M		0·8600	136-142	85	6	3	6

Specific gravity of the paraffins, 0·7407.

I must here mention that in all determinations of the distilling degrees of commercial xylene the thermometer was in the liquid—the bulb about half an inch from the bottom of the retort—which practice I would always advise when dealing with mixtures of hydrocarbons or their derivatives. The paraffins separated from a number of samples of crude xylenes distilled, thermometer in liquid, from 145° to 151° C.

I have to thank Mr. George E. Davis for a sample of crude xylene which he obtained by fractional distillation from crude gas benzene. The distilling degrees of this were 138° to 141°. It was interesting to determine the composition of this crude gas xylene, and I give you the result as follows: Metaxylylene, 47 per cent; paraxylylene, 8 per cent.; paraffins, 26 per cent.; compounds oxidisable by dilute nitric acid, 19 per cent.; total, 100. Unfortunately, I had not a sufficient quantity to regenerate the orthoxylylene from its sulpho-salt; but from the amount of ortho-salt obtained, I estimate approximately that of the 19 per cent. oxidisable compounds 13 per cent. were orthoxylylene. The metaxylylene, paraxylylene, and paraffins were determined by methods A and B. I account for this sample of crude gas xylene containing such a large amount of paraffins by its high boiling degrees, and I believe that if Mr. Davis had collected the fraction distilling between 136° to 141° the result would have been a higher percentage of metaxylylene and less paraffins. I beg you finally to understand that I do not claim for these methods, which I have based on O. Jacobsen's and A. Brückner's investigations, absolute scientific accuracy, but I have found them to give very satisfactory results for all practical purposes. I have converted many thousand gallons of xylene into metaxylydine, and have thus had the opportunity of practically establishing in some measure the correctness of these methods, and I hope that the communication of them may be as serviceable and valuable to members interested in this subject as they have been and yet are to myself.

\*Instead of converting these sulpho-acids into their sodium salts, it might be preferable to produce the barium salt or other salts.



## DISCUSSION.

MR. WATSON SMITH: With regard to the valuable communication of Mr. Levinstein, I think he seems disposed to credit himself with too little. Jacobsen and Brückner had worked out certain methods for separating the isomeric xylenes, in order principally to obtain preparations, and hence merely aimed at what may be termed qualitative separation. They did not attempt to discover by what combination of means a tolerably expeditious method of quantitative separation might be built up, to be used in the estimation in mixtures of the proportionate quantities of the isomeric xylenes and paraffins present together. Mr. Levinstein, whilst pointing out that only the metaxylene is useful to the colour manufacturer, has also indicated that by means of a suitable combination of parts of the methods of Jacobsen and Brückner useful and expeditious methods of determining the quantities of the isomeric xylenes in mixture together may be found. About a week ago Mr. Levinstein placed his method in my hands, and I undertook to test its efficacy on some samples of commercial xylenes also sent me by Mr. Levinstein, which samples he had previously tested by the process. I handed the samples over to Mr. Alfred Rademacher, one of the students in the laboratory of the Owens College, and gave him the necessary directions, and he has not found the least difficulty in carrying out the method. The following are the figures obtained:—

		Meta- xylene.	Para- xylene.	Ortho- xylene.	Para- ffins.
I. Sample of xylene (2 determinations) (1)	87	1	2	3	6
	86½	1½	2	3	6
II. Sample of English xylene..... (2)	81	3	1½	1½	1½

[Mr. Levinstein here pointed out that Sample I. corresponded to that marked L in his table, and Sample II. to E in the same table, which see.] In the estimation of the metaxylene, where the alkaline solution is distilled with wet steam, to separate the unaltered meta-isomer and paraffins from the alkaline solution of toluic acids, etc., in one experiment the volume of the naphtha was measured before steaming. It was then put back, and the mixture steamed, two Liebig's condensers, each about a yard long, being joined together to insure good condensation. The naphtha condensed was again measured. Volume before steaming = 97 c.c., and after steaming = 95 c.c.; hence but little loss was sustained in the distillation. In the treatment with sulphuric acids of different strengths it is, of course, necessary to shake repeatedly with fresh quantities of acid, in a suitable stoppered tap-funnel, until no change of the volume of the naphtha occurs after the last shaking with acid. To a certain extent the fact of the fuming acid remaining clear and colourless after the last shaking, in the estimation of paraffins, may be taken as an indication that all or nearly all the xylene is removed. When, moreover, the xylenes are thoroughly removed, and only paraffins remain, if a drop of these paraffins be allowed to fall on the back of the hand and be spread with the finger, a smell resembling that of petroleum spirit will be observed. Little more than a trace of xylenes would communicate the characteristic odour of that aromatic hydrocarbon to the paraffins. Turning now to Mr. von Hohenhausen's paper, I note that he puts the specific gravity of 90 per cent. benzol at 0.882, and Scotch 90 per cent. at 0.870. As an exceptional case I may mention that of a 90 per cent. benzol from a London gasworks, which I found to contain 6 per cent. of carbon bisulphide—the result of possibly very high carbonising temperatures on a pyritous coal. This benzol had a specific gravity of 0.900. The nauseous odour of the carbon bisulphide was completely covered and masked by the faint odour of the benzol—a somewhat

curious fact. After removing the  $CS_2$  the specific gravity of the benzol became 0.880. Although the specific gravity of Scotch 90 per cent. benzol be 0.870, reduced by the presence of paraffins and other bodies of low specific gravity, yet I could understand the presence of carbon bisulphide even in such a naphtha; for the effect of the high specific gravity of this substance on the one hand might be much more than counter-balanced by the very low specific gravity of a due proportion of paraffins also present, the specific gravity of these light paraffins varying from 0.630 to 0.700 about. I presume even Scotch crude coal gas contains carbon bisulphide, and hence I hardly see how the first runnings of the benzol from the Scotch tars can escape the presence of at least some little of the bisulphide. A quick as well as a really accurate method of estimating the amount of carbon bisulphide in benzols would be a boon. I have a sample of vinic alcohol sent me by my friend Dr. Otto N. Witt, which was extracted from commercial benzene in Germany, and Dr. Witt informed me that so considerable is the amount of alcohol that its extraction as a by-product was proposed as a profitable undertaking. There were only traces of alcohol in the sample of London benzol I examined. If I were to ask any member present to smell this sample of alcohol from benzol, (it contains traces of nauseous sulphur compounds), he would be very sorry, by way of experiment, to taste a portion of it.

MR. TRUBY: I think what Mr. Hohenhausen has said with regard to the distillation of benzol is correct, but I must take exception to some of the specific gravities mentioned by Mr. Hohenhausen. I have worked Scotch benzol of as low specific gravity as .860. In all other respects I quite agree with Mr. Hohenhausen. You can depend more on the English than on the Scotch benzol, as it gives better results. I think it would be very difficult to adulterate with bisulphide of carbon, on account of its high specific gravity and other properties. I have worked samples which contained as much as 12 per cent. unnitrifiable products, and yet possessed a high specific gravity. I have also observed that some of the lighter boiling portions, distilling below 80°, have sometimes had a greater specific gravity than the original benzol.

MR. DREYFUS said researches had been made and published where the separation of ortho-, para-, and meta-xylenes had been investigated by Dr. Jacobsen and Dr. Brunck, and it was of the highest importance that the users of xylol should know the proportions of the various isomeric xylenes which were contained in the commercial article. Great credit was due to Mr. Levinstein for having brought the subject before them. With regard to Mr. Hohenhausen's paper on the distillation of crude naphtha, I must say that I am rather disappointed. Mr. Hohenhausen seems to have skipped the most important part, that of the washing process previous to rectifying. I was under the impression that we should have heard of processes in the paper which were not generally known, whereas, to my great disappointment, no such new matter has been brought forward. Most of the apparatus which has been shown to us on the screen, if not all, is contained in a book published about two years ago by Dr. Schultz, of Strassburg, and there is no doubt that the apparatus described in that book would be useful to tar distillers and rectifiers of benzol. As far as the quantity of bisulphide of carbon is concerned, the same exists in large quantities in the London benzol and in less quantity in Midland benzol, whereas the Scotch benzol is quite free from it. Mr. Nickels, of London, has devised a test whereby the quantity of bisulphide of carbon contained in benzols can be ascertained. He mixes the benzol

to be tested with some caustic potash and absolute alcohol, shakes it up for a certain length of time, and allows it to stand, when xanthate of potash is formed, which separates out and can be weighed. With respect to Mr. Truby's remarks, that he found some 90 per cent. Scotch benzol to have a specific gravity of '860, I state most emphatically that I have never met with such a low specific gravity stuff, and if I had met with it I should have looked upon it as being adulterated, and not as genuine benzol. If such benzol, as stated by Mr. Truby, were submitted to nitrification in the usual way, considerably over 5 per cent. of matter would have been found that would not nitrify. The average specific gravity of the Scotch 90 per cent. that has come under my notice has been '870 to '875, and of 50 per cent. Scotch benzol '867 to '872. As far as the specific gravities of the English benzols, as given by Mr. Hohenhausen, are concerned, I think they are correct. I cannot understand the question put at the end of Mr. Hohenhausen's paper, in which he says: "What becomes of the stuff distilling between 40° and 80°, 82° and 110°, and 110° and 136° C.?" These products are not worthless, as they contain still a certain quantity of toluol and xylol, and anyone conversant with distillation will know that if such naphthas are rectified or distilled over again a further quantity of xylol is obtained.

Mr. H. GRIMSHAW: There is one aspect of the question of the increased demand for xylene, which I think is worth bringing before the notice of this meeting, as it closely affects the interests of a very large and important industry in this country and in other parts of the world. I allude to the indiarubber manufacture, in which solvent naphtha is very largely consumed. What is called solvent naphtha at one time consisted, I believe, of all those portions of the rectification of light coal-tar oils which distilled above benzol, and below about 160° C. Already for some years it has been the practice to abstract for the aniline manufacturer the toluol boiling at 110° and over, and now the demand for xylene is evidently causing the product immediately above toluol to be also abstracted. It seems to me that it will become a question for the indiarubber manufacturer to determine with exactitude what particular hydrocarbons are most valuable for the solution of indiarubber, and this subject presents, without doubt, a field for valuable and interesting research on the part of any chemist who may have the opportunity of taking it up in a scientific manner. The question of the proper solvent for an article which in the form of fine Para rubber is worth from four to five shillings per pound, and is annually imported into Liverpool alone to the value of nearly two million pounds sterling, is a very large question, and will well repay the labour devoted to it. Allusion has been made to the anomalous character of many commercial benzols of apparently correct boiling point, caused by the presence at one and the same time of carbon bisulphide and light paraffins. This is only what might be expected when we consider the fact that the boiling point of chemical substances is not at all a function of their specific gravity; that though heavy liquids do as a rule boil at a higher temperature than light ones, still there are many comparatively heavy liquids which boil at low temperatures. Carbon bisulphide, chloroform and bromine are instances of this. The naphtha adulterator is therefore in the same happy position as the milkman, who by judicious abstraction of cream and addition of water can arrange his milk to have exactly the same specific gravity as before the sophistication. Facts such as the above therefore render much more valuable such methods of determining the value of liquid products as that brought forward by the chairman, for a precise chemical method of valuation must

almost always be superior to what may be called a superficial reliance on the physical properties of boiling points, specific gravity, and so on.

Mr. GEORGE E. DAVIS: I have failed to gather from the remarks of the previous speakers what actually becomes of the intermediate products other than benzene, toluene, and xylene. It must be evident that if these products go back again and again into the still, and pure benzene, toluene, and xylene only run off, that these compounds must accumulate. These compounds, consisting as they do of paraffins and olefines, although in small quantity, may exert an injurious influence in colour manufacture. Mr. Truby has referred to the fact that some of his lighter distilling portions possessed a higher gravity than the original benzol. This may be due to the fact that the distillate was a mixture of bisulphide of carbon with substances possessing a lower boiling point than benzol, probably paraffins, and the former being in a greater proportion than exists in benzol would raise the gravity. I have been very pleased to hear Mr. Levinstein's paper upon the subject of the determination of meta-, para-, and ortho- xylene, and especially to find that there was a large quantity of orthoxylylene in the sample of xylene which I separated from coal gas. Not being an expert in these matters, I was rather troubled on receipt of a letter from Professor Armstrong stating that orthoxylylene did not usually exist in English light oils, but Mr. Levinstein has placed upon the black-board the analyses of many samples of commercial xylene in which the existence of orthoxylylene is placed beyond doubt. Mr. Grimshaw has raised a most important question relative to the future commercial value of solvent naphtha. There have been many complaints amongst the indiarubber goods manufacturers lately regarding the quality of solvent naphtha now in the market, and it seems, therefore, to point to this, that the abstraction of xylene deteriorates its quality for the manufacture of rubber goods. It is open, therefore, for chemists to either find a substitute for solvent naphtha which will serve the purposes of rubber goods manufacturers, or else tar distillers will have to leave the xylene in the solvent naphtha, as before.

#### REPLY.

The CHAIRMAN: Mr. Hohenhausen, who unfortunately cannot be present this evening, has asked me to answer, as far as I am able, any question which might be raised in reference to his paper. I must confess that I feel rather embarrassed in performing such a task, seeing this evening several of the most experienced refiners amongst us. In reference to Mr. Dreyfus's remarks, I am sorry to hear that he was disappointed with Mr. Hohenhausen's paper, but I must tell him that he is mistaken when he states that the different apparatus described by Mr. Hohenhausen may be found in Schultz's most excellent work, "*Die Chemie des Steinkohlentheers*." I certainly do not recollect that this very useful book gives a description of Vedles' two forms of apparatus for separating benzene and toluene, nor of Egrot's dephlegmator, nor of Aders' (Magdeburg) valuable refiner. Dr Schultz's book has not been translated into English, and if Mr. Hohenhausen had even taken some illustrations from this book, I fail to see, gentlemen, that such an abstraction would in any way have depreciated the merits of his paper. In reference to the washing of crude naphtha—which operation does not really come strictly within the subject of the paper—I think that Mr. Hohenhausen has in some measure explained it, but if this explanation is not satisfactory to Mr. Dreyfus, that gentleman might do great service to our Society by favouring



us at some future time with a paper on the subject which appears to him to be of so much importance. Mr. Dreyfus thinks that Mr. Truby's 90 per cent. Scotch benzene of '860 gravity must have been adulterated; but is it not possible that the difference in the temperature at which the samples were tested might to some extent account for the difference in the specific gravity? There certainly are some intermediate products in the fractions distilling between 40° to 81° C. and 111° to 134° C. which are neither benzene, toluene, nor xylene, and it would be no doubt very interesting, and valuable to find some proper use for them. I now come to my own paper, and I am glad to hear that Mr. Watson Smith obtained with my method similar results to those shown to you on the blackboard. Mr. Dreyfus, if I rightly recollect, stated that Messrs. Jacobsen and Brunck's investigations have already led them to describe methods for the separation of ortho- meta- and para- xylene, but I am not aware that Brunck has published any communication having the slightest reference to the separation of xylene. Mr. Dreyfus probably means Brückner, a chemist whom I have already mentioned in my paper—but neither he nor Jacobsen have endeavoured to found a quantitative method for determining the commercial value of crude xylene. In reference to Dr. Armstrong's communication to Mr. Davis, I am surprised to hear that the former gentleman scarcely ever found orthoxylene in English light oils. My own experience has convinced me that crude xylene, obtained from English naphtha, is never without orthoxylene. At least in the large number of samples of crude xylene which I have hitherto examined, I have always found orthoxylene to be present, and I presume that if crude xylene contains orthoxylene the latter must also have been in the light oil. In reference to the olefines, which have been mentioned, I believe by Mr. Davis, I beg to say that these hydrocarbons occur more abundantly in crude gas benzene, and also in Scotch naphtha, than in English naphtha, at least the quantity of olefines or non-saturated hydrocarbons in crude English xylene is generally very small. I quite agree with Mr. Grimshaw, and it might be interesting to him to know that solvent naphtha, after abstracting from it the xylene, is deteriorated in quality, at least as far as the rubber manufactures are concerned. I have heard of a case in point where a large quantity of solvent naphtha had been returned by a rubber manufacturer on account of the abstraction of xylene. I think that if refiners abstract the xylene from the solvent naphtha it might perhaps be advisable to replace it with toluene, and I also think that the methods described in my paper for determining the value of crude xylene might be made applicable, with a few modifications, for determining the value of solvent naphtha.

## Newcastle Section.

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Meetings—Session 1884: March 6th, 1881.

GENERAL MEETING AT THE COLLEGE OF SCIENCE,  
NEWCASTLE, February 7th, 1884.

## ON SOME IMPROVEMENTS IN THE ESTIMATION OF TANNINS.

BY HENRY R. PROCTER, F.C.S.

Of all the methods which have been proposed for the estimation of tannins, the only one which has met with any general acceptance is that of Löwenthal, and indeed it is the only one which in rapidity of execution and constancy of results is fitted for general use. The method, as originally proposed, depends on the oxidation of the astringent solution by permanganate in presence of indigo, which not only serves as an indicator, but controls the oxidation, limiting it to those bodies which are more oxidisable than indigo. As, however, these include gallic acid and other substances which are useless to either tanner or dyer, it is necessary to remove the tannin, and by a second titration to obtain its value by difference. This Löwenthal (*Zeitschrift für Anal. Chemie*, 1877, p. 33) accomplished by a solution of gelatin and common salt, to which, after mixture with the tannin infusion, a small quantity of sulphuric or hydrochloric acid was added. It was necessary to let this stand at least some hours before a clear filtrate could be obtained, and the gelatin remaining in solution had a slight though generally negligible effect on the permanganate. In some cases, even after long standing, perfect filtration was extremely difficult and tedious, and it was also clearly proved by Simand (*Dingler's Polyt. Journal*, bd. 244, s. 400) that a certain proportion of the tanno-gelatin precipitate, varying with the acid present, and with the species of tannin, remained in solution, and thus gave too low a result. He therefore proposed to revert to the old method of separating tannin with hide raspings, or, as an improved substitute, with the gelatinous tissue of bones. This, however, though accurate when carried out with extreme care, is both a tedious and difficult process, and quite unsuited for general technical use. I have therefore tried, and I think successfully, so to modify Löwenthal's original method as to increase its accuracy, and at the same time to make it more rapid and easy of execution. I found that by saturating the clear filtrate with salt, a further precipitate containing tannin was formed, but, unfortunately, it was so finely divided that no amount of standing, or even of warming, and repeated passing through the paper, would obtain a clear filtrate. Finally, I hit on the device of mixing with the liquid, before filtration, a portion of the pure kaolin used by photographers. The effect was instantaneous and complete. A perfectly clear filtrate was obtained without any of the tedious waiting which before was necessary, and it was not only free from tannin, so far as I have been able to ascertain, but also nearly so from gelatin, so that it only gives the faintest cloudiness with tannin solution. Gelatin gives a more considerable precipitate, but this is simply due to its insolubility in the saturated salt solution, and it is redissolved on dilution with water.

I now generally work in the following manner: I employ permanganate of the strength of 1grm. per litre and solution of the purest indigo-carmin of 5grms. with 50c.c. of concentrated sulphuric acid per litre, using a 25c.c. burette and 20c.c. of indigo solution, which consumes about 15c.c. of permanganate. The quantity of astringent used must not require more than the remaining contents of the burette. The titration is performed in a white basin as recommended by Kathreiner, with about  $\frac{3}{4}$  litre good water, which it is best to measure approximately, so that if it con-

tains any impurity which affects the permanganate it should be constant, and thus be eliminated with the indigo. The titration is finished when the pure yellow liquid shows a faint pinkish rim. This end-reaction, which is of extraordinary delicacy, is due to Kathreiner, and is quite different to the pink caused by excess of permanganate, being an effect common to all pure yellow liquids. I do not find it needful to make the titration so slowly as has been advised—the permanganate may be dropped in rapidly, with vigorous stirring, so long as there is large excess of indigo, but as soon as the bottom of the basin can be seen through the solution it must be added very cautiously, and with occasional pauses, to allow time for its complete mixture through so large a mass of fluid. I make my infusion of such a strength that I can employ 5c.c. of the original liquid for each titration. This is repeated twice, and the results added together and denoted by *a*. I then take 50c.c. of the infusion, and add 28.6c.c. of a freshly-made solution of Nelson's gelatin of 2grms. to 100c.c. After shaking the mixture is saturated with salt, which brings the volume up to 90c.c., and 10c.c. of dilute sulphuric acid (containing 1 vol. of concentrated acid in 10) and a teaspoonful of pure kaolin are added. It is best to do this in a flask in which it can be well shaken, after which, filtration may be at once proceeded with (the flasks may be cleansed with caustic soda solution). 10c.c. of this filtrate (= 5c.c. of the original infusion) are employed for a second pair of titrations, which are added as before, and the result denoted *b*. If, further, *c*, be the quantity of permanganate required to oxidise 10c.c. of decinormal oxalic acid, and 10grms. of substance have been employed to 1 litre of infusion,  $c : (a - b) :: 6.3 : x$ , where *x* is the percentage of tannin expressed in terms of crystallised oxalic acid. For the present I invariably calculate my results in this way, since we do not actually know the relation of any single tannin to permanganate, even Neubauer's number for gallotannic acid being probably too high, and Oser's for quercitannic being only a fair approximation. It happens, moreover, that this last equivalent (62.36) does not differ from that of oxalic acid (63) more than the ordinary limits of error of such estimation, and the substitution is therefore of no commercial importance, while it is surely better to employ a standard which is easily and exactly verified than one which is certain to be modified by further research, and so to run the risk either of having our results made useless for future comparison, or of establishing a false and arbitrary equivalent. What is wanted for practical purposes is not the absolute weight of tannins in the various materials, but only a means for the relative comparison of two samples of the same material; cross comparisons of different tannins being simply delusive. If, however, it is necessary at any time to give actual percentages of gallotannic acid, it is probably best to stick to Neubauer's number for the present, as it is in general use and as likely to be correct as any other. I think when this is done the equivalent used should be definitely stated, or it will certainly lead to confusion. Neubauer's equivalent is only properly applicable to gall nuts, and possibly to sumac and myrabolans. For oak bark Oser's number or that of oxalic acid is most likely nearly correct; and this may also be approximately true of oak wood and valonia, but as respects all other materials we have no information whatever, and the oxalic equivalent is as likely to be right as any other.

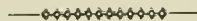
I have been unable from lack of time to make so complete an examination of the results of the process with various materials as I should have liked, and as I hope to do later; but in every case in which I have

tried it, the results have been sharp and concordant, and there has been no difficulty in filtration, even with those tannins which before were most apt to give trouble. I give below a few results, not as showing the relative values of the materials, which, of course, cannot be directly compared by any analytical process, but for comparison with those obtained by other methods and modes of calculation:—

Spent Liquor .....	Tannin (as oxalic acid).		Other bodies oxidised (as oxalic acid).	
	0.12	.....	11.0	.....
Valonia (good Smyrna).				
Sample 1.....	29.1	.....	2.3	
Valonia (good Smyrna).				
Sample 2.....	30.7	.....	2.1	
Valonia (good Smyrna).				
Sample 3.....	30.5	.....	1.9	
Hungarian Larch Extract.				
Sample 1.....	14.78	.....	1.95	
Hungarian Larch Extract.				
Sample 2.....	18.08	.....	2.33	
Chestnut-wood Extract,				
25" B.....	25.33	.....	3.68	
Pegu Cutch.....	63.50	.....	2.45	

I have proved by experiment that kaolin removes nothing which is oxidised by permanganate, but simply facilitates the precipitation and filtration; and I have often found it useful in clarifying the original infusions and liquors before the first titration. On the other hand, there is no doubt that the salt and acid of Löwenthal's method precipitate of themselves a large proportion of certain tannins. In the case of cutch this amounted to 67 per cent. of the whole. I think, however, there is good reason to believe that these bodies would also have been absorbed, or at least removed from solution by hide in the process of tanning. This is shown by the analysis of spent liquor, which originally contained the tannins of oak bark, valonia, myrabolans, gambier, hemlock, and, oak-wood extracts, etc., to the extent of 10 to 15 per cent., but which was reduced by contact with hide to 0.12 per cent. That a portion had not been absorbed but decomposed is proved by the large accumulation of oxidisable impurities (equal to 11 per cent. of oxalic acid); at the same time this example shows that the method is capable of estimating a very small portion of tannin in presence of much gallic acid and other analogous substances. It is worth remark that such spent liquors become very pale in colour, and also that the filtrates, freed from tannin by precipitation, are nearly colourless, thus proving that the colouring matters present in tanning materials are of the nature of tannins, at least as regards their precipitability by hide and gelatin.

It would be out of place here to enter upon the methods of sampling and grinding tanning materials for analysis, though I believe this to be frequently one of the most difficult parts of the process. With regard to extraction, I may mention that I have found brisk boiling in a large flask with a litre of water for half an hour to be more effective as well as less troublesome than repeated exhaustion with smaller quantities. In dissolving extracts it is well to pour them into water actually boiling, as in this way uniformity is attained, many extracts becoming permanently insoluble if mixed at first with water at too low a temperature.



## CONTRIBUTIONS TO THE DEVELOPMENT OF THE SPRENGEL AIR PUMP.

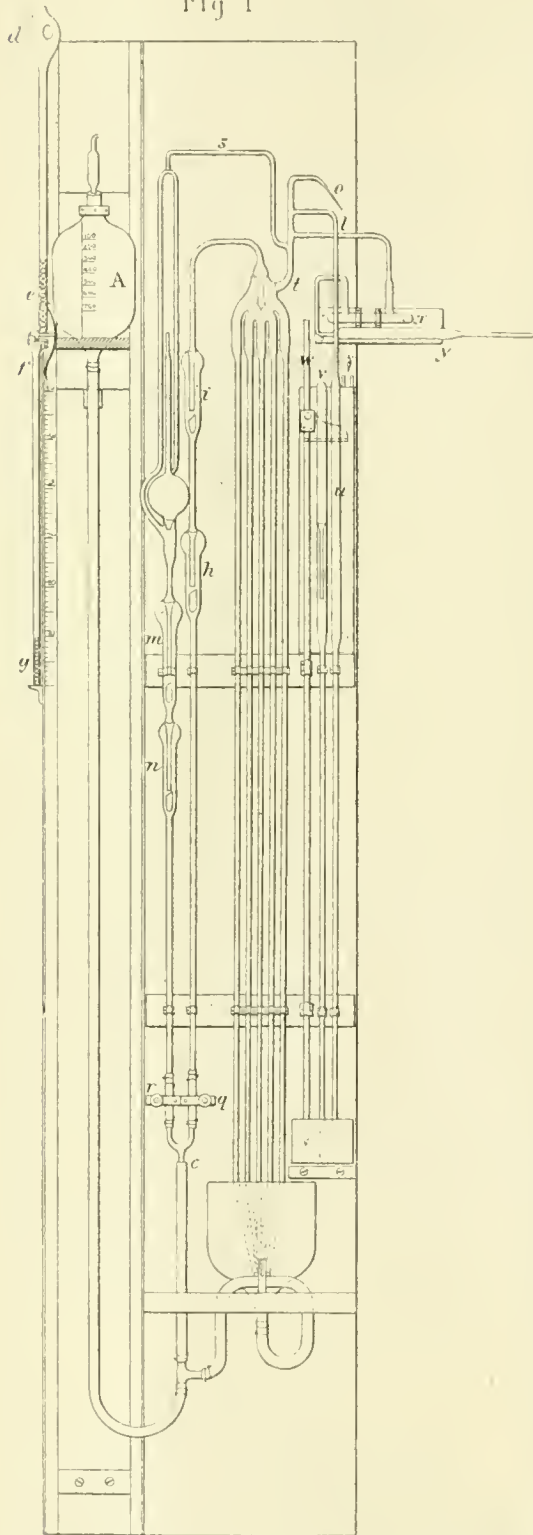
BY CHARLES H. GIMMINGHAM, F.C.S.

AT the close of the year 1876 I wrote a short paper, which was published in the Proceedings of the Royal Society, on a modification of the Sprengel air pump, and showed that the slowness of the process of exhaustion, which had always been a drawback to



the employment of that air pump, might be, to a great extent overcome by the adaption described in

Fig 1

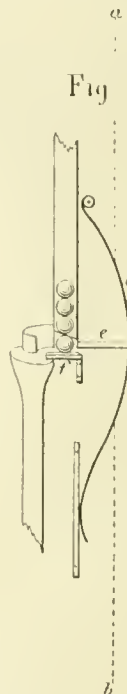


my paper. This modification is now well known, and has come into pretty general use. The result of con-

tinued experiments directed towards a similar end has been so far successful that I think I am justified in inviting for a second time the attention of the scientific public to the subject. I will now endeavour to point out, as briefly as the subject will permit, what the alterations are which I have to suggest, what results they will effect, and by what means and from what causes those results are produced; and I shall further add a few observations relating to the working of the instrument, which may be useful to those who have but small practical acquaintance with it.

The principal objects kept in view in my later efforts were those of accelerating the process of exhaustion, and of rendering that exhaustion more complete; but in addition to this it seemed to me exceedingly desirable to construct some form of jet which could be adapted in a simple way to a multiple fall tube, and before proceeding to discuss the rate of exhaustion with fall tubes of various diameters it will be well to give my reasons for adopting the par-

Fig 2



ticular form of jet that I use. From time to time various jets have been devised, mostly with the idea that the mercury should fall regularly and in long pistons down the fall tube. To my mind this is by no means necessary to the successful working of a pump, and it prevents all possibility of obtaining a rapid exhaustion. The advantages possessed by my form may be briefly stated as follows: The simple way in which it can be adapted to a multiple fall tube pump. In producing high exhaustions the great variation in the speed of the mercury flowing down the fall tubes that it affords is exceedingly useful, as it is often good at the higher exhaustions to pass the mercury through alternately slow and then fast. The mercury running slowly collects bubbles of air just below the point where the hammering takes place, and then a fast stream will generally carry them out, or far down the tube, to await the arrival of other bubbles until they are swelled sufficiently to be carried out.

Before proceeding further it will also be well to glance at and dismiss an addition to the instrument

of minor importance, but whose usefulness and simplicity of construction demand a few words of explanation. In experimenting I often found it necessary to count the number of times the mercury reservoir was raised, in order to calculate the amount of mercury passed through the pump, and for this purpose the shot counter shown on the left-hand side of the reservoir A in Fig. 1, and enlarged in Fig. 2, was constructed for automatically registering each bottle of mercury. Two tubes, *d e* and *f g* (Fig. 1), are placed one above the other. The upper one has a small bulb at the top with an opening in it through which shot may be inserted. The shot should be picked all of one size, and just loosely fit the tubes. The lower tube is closed at the bottom and blown into a funnel

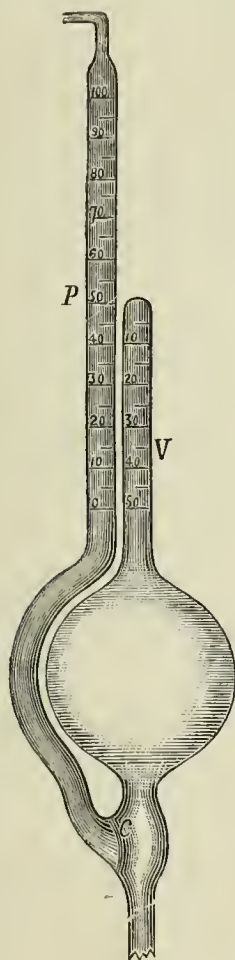


Fig. 3.

shape at the top to receive the shot as they are transferred from the upper tube, one by one, each time the reservoir A is raised. The shot being all of one size a scale may be made to indicate without counting the number of times the reservoir has been raised. The tube *f g* when filled can be removed, and the shot poured back into the opening in *d*. The arrangement for transferring the shot one at a time from the upper to the lower tube will be easily seen by reference to Fig. 2. The position that the slide carrying the reservoir A takes when raised is shown by the dotted line *a b*. It will be seen that the spring *c* will be pressed inwards, which will force, by means of the projecting piece *e*, one shot over the little brass support *f* and *e*, remaining in its new

position as long as the reservoir is raised, thus preventing the next shot from falling down. As soon, however, as the slide is lowered the spring draws *e* out, and a new shot comes down ready to be forced out the next time the slide comes up. The one I have counts to 100, which is more than I have ever required. In order to divide the mercury into smaller portions than the total contents of the bottle it is graduated at every 100c.c., the whole contents being 800c.c.

I shall now consider a more important branch of my subject, and ask you to follow me through a series of experiments all pointing to the conclusion that perfection of the instrument is only to be

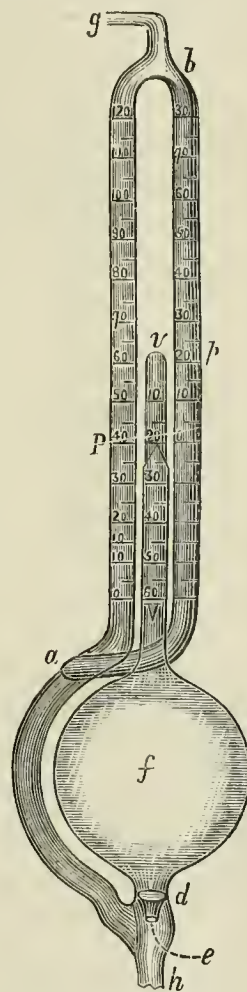


Fig. 4.

attained by a careful regulation of diameter and length of the fall tubes employed.

It will perhaps be the more explanatory as it is the more natural plan that I should narrate the experiments in about the order in which they took place, premising that the tabular statistics appended, and to which I must often refer you, are extracted from notes carefully recorded at the time of the respective experiments.

In order to accelerate exhaustion I increased the number of fall tubes from 3 to 5, and in one pump that I used in my own laboratory for a long time, to 7. Although the results with the seven-tube pump were quite satisfactory, yet I found that for general use



a three or five tube pump was preferable. In the case of the seven fall tubes I used four of comparatively large diameter for rapidity, and three smaller ones to get the higher exhaustions not easily accomplished with fall tubes of large diameter. The rate of exhaustion with this pump was very great for one constructed on the Sprengel principle, but the supply of mercury also had to be very large, a bottle containing 800c.c. only serving for three or four minutes, which necessitated so much lifting and waste of time (the transference of the mercury from the lower reservoir into the movable one occupying almost as much time as its passage through the pump) that I afterwards used two small fixed reservoirs and transferred the mercury from the lower one to the upper by means of a small force pump, which was constructed out of a stout glass tube, using boxwood and leather for the plunger and valves. The pump could then be worked by the circulation of a few pounds of mercury, and had one sufficient work to keep a gas engine going on the force pump a great advantage would be gained. For laboratory work, however, I came to the conclusion that I had done too much in the direction of rapidity, as it will be seen further on that exhaustions cannot be reproduced

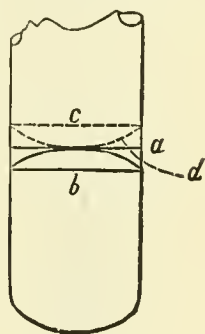


Fig. 5.

at any desired rate unless the connection between the pump and the apparatus to be exhausted is very short and open, two conditions very often impossible to obtain. To ascertain the best length and diameter for the fall tubes a single fall tube was arranged in front of my five-tube pump, a general view of which is given in Fig. 1. The single fall is not shown, as it was only temporarily arranged. It, however, was supplied from the reservoir A by making an extra joint in the glass YY-piece at *c*, and arranging a third pinch-cock there similar to *r* and *q*. The mercury passed from the reservoir through the supposed joint at *c* (Fig. 1) and two ordinary air-traps exactly similar to *h i*, which have been already described in my previous paper, but in which I have since introduced a slight improvement, consisting of a piece of twisted iron wire placed in the inner tube of the trap, which prevents the mercury forming pistons, and thus facilitates the emptying of the traps into the vacuum, when it is desired to re-exhaust them. The fall tube was attached to the jet by means of a stopper which was fixed by vacuum cement.\* An arm from the upper wide part of the fall tube was connected to the five-tube pump at *l*, thus connecting the two pumps together and avoiding the necessity for separate drying tubes and measuring apparatus.

The first point—viz., the best length for the fall tubes—was decided some time back to be 39in., measured from the average height of the mercury

in the lower reservoir. This length has been arrived at from practical experience and making careful notes on the working of each pump that I have from time to time fitted up. If the fall tubes be too short the pump works very slowly, owing to the column of mercury (deducting the air spaces) not being of sufficient length to run quickly down the tube, thereby occasioning a constant filling up at the top end and consequent waste of mercury power. An experiment recorded in my note-book with a five fall tube pump whose tubes were at first made 33in. long, and then were lengthened to 39in. by sealing pieces to their lower ends, shows that by passing the same quantity of mercury through at the same rate, a certain globe was exhausted in the first case to 50mm. pressure, and in the second to 1mm., showing a great increase in the rate of exhaustion, due to the extra 6in. of fall tube. On the other hand, if they be made a greater length than 39in., the fall of the mercury at the high exhaustion causes such severe hammering that the tubes are liable to be fractured. In testing the different sized fall tubes the same length was adopted in each case, and also the same cubical contents to be exhausted. The latter was ascertained by collecting the gas at the end of the fall tube and carefully measuring it, the result being 136c.c. The first fall tube experimented with was a very wide one, in which the mercury was only just able to form pistons.

The following experiments were tried with the double object of finding the best size, and also the most economical rate for the mercury to flow through the jet. It will be seen from table 1 that it took 2,500c.c. of mercury, running at the rate of 100c.c. in 1·2 minute, to accomplish the same work that was done by 1,600c.c. of mercury running at the rate of 100c.c. in 5 minutes. On the other hand, we have the time saved by using the mercury at the quick rate, equal to 50 minutes.

FALL TUBE No. 1, 2·4mm. DIAMETER.

c.c. of Mercury.	Pressures in millimetres recorded with mercury running at the rate of	
	100c.c. in 1·2 minute.	100c.c. in 5 minutes.
100	329	368
200	236	242
300	186	187
400	146	133
500	126	108
600	106	85
700	85	72
800	73	55
900	69	46
1000	57	33
1100	50	24
1200	41	16
1300	36	10
1400	29	6
1500	21	3
1600	20	1
2500	1	—

This tube being too large for ordinary use, further experiments were not tried with it, except at higher exhaustions, of which I shall speak presently. It will perhaps be noticed that the exhaustions produced by the slow rate at first are less than those by the fast rate. This discrepancy is due to loss by the mercury running down the tube without properly forming pistons, owing to the large diameter of the tube. The amount of this loss will be seen by referring to the table giving the results with fall tube No. 2, which is of smaller diameter, yet we have the same

\* A mixture of beeswax and resin : 3oz. beeswax, 3oz. resin.

amount of work done by 700c.c. of mercury that it took 1,600c.c. of mercury to do with the large tube No. 1, the rate of flow and contents to be exhausted being the same in both cases.

FALL TUBE No. 2, 1.8mm. DIAMETER.

c.c. of Hg.	Pressures recorded with mercury running at rate of	
	100c.c. in 5 mins.	100c.c. in 2 mins.
100	232	249
200	128	119
300	79	87
400	39	61
500	20	36
600	8	22
700	1	16
800	—	8
900	—	6
1000	—	4
1100	—	2.5
1200	—	1

This table also shows a gain in time by allowing the mercury to flow fast, but not bearing anything like the proportion to the amount of mercury expended which was shown with No. 1 tube. In both cases, however, extremes have been taken, and the most economical rate will be between the two experimental times taken. The experiments with No. 3 fall tube show how largely the rate of exhaustion of different pumps may vary by having differences in the diameter of the fall tubes, which from the outside would be scarcely visible.

FALL TUBE No. 3, 1.4mm. DIAMETER.

c.c. of Hg.	Pressures recorded with Mercury flowing at rate of	
	100c.c. in 10 mins.	100c.c. in 4 mins.
100	470	582
200	341	461
300	258	358
400	197	295
500	155	249
600	119	208
700	93	178
800	80	153
900	66	132
1000	50	116
1100	38	102
1200	29	86
1300	23	73
1400	15	65
1500	10	56
1600	5	48
1700	3	42
1800	1	33
1900	—	26
2000	—	21
2100	—	17
2200	—	13
2300	—	10
2400	—	6
2500	—	4
2600	—	2.5
2700	—	1

This tube, it will be seen, took 108 mins. to produce the same exhaustion that was done in 24 mins. by the previous tube, with a far less expenditure of mercury power. In this table we have the same thing shown as in No. 1, viz., a large increase of time saved at the expense of mercury power. With this tube No. 3 an experiment was tried with the mercury running at the rate of 100c.c. in 30 mins., and producing an exhaustion of 1mm. pressure with 1,500c.c., whereas by using 1,800c.c. of mercury the same work was done in 3 hours, and 2,700c.c. accomplished the same exhaustion in 1 hour 48 mins. Although I considered this tube small, and there was a good deal of mercury

power wasted by the filling up of the top part of the fall tube, I thought it would be well to try a still smaller tube, more especially for the sake of the results at higher exhaustions.

FALL TUBE No. 4, 1.1mm. DIAMETER.

c.c. of Hg.	Pressure recorded with mercury running at rate of 100c.c. in 5 mins.
300	361
600	263
900	195
1200	145
1500	110
1800	86
2100	67
2400	52
2700	34
3400	4
4000	1

Table 4 shows the result up to 1mm. pressure. The maximum speed at which the mercury could be run with this tube was 100c.c. in 15 mins., and it will be seen that 10 hours were occupied and 4,000c.c. of mercury. This enormous increase of mercury used is caused entirely by waste, on account of the incessant filling up of the wide part of the fall tube, the direct cause of which is the friction due to the small bore of the tube.

Owing to the very limited time that I have had at my disposal for original work, I have not been able to carry out the experiments at higher exhaustions with these single fall tubes to the same detailed extent as those at the lower exhaustions. However, I have arrived at very definite conclusions with regard to the sized tubes that should be adopted for producing high exhaustions. At one time I had an idea that if a pump could be so constructed that as soon as the rarefaction had reached the barometric height by exhaustion through large fall tubes, the mercury could be turned so as to exhaust through a set of fine bore tubes, I should be able to get a high vacuum easily, because the pistons of mercury, being of small diameter, would enclose bubbles of rarefied air which would escape in a larger tube. On experimenting with pumps constructed on this principle, I soon found that I could produce as good an exhaustion with the large tubes alone as I could by using the finer ones at higher stages. It is therefore not a case of producing pistons at the higher exhaustions, the last portions of gas being taken out by a process of entanglement. The contents being exhausted remaining at 136c.c. the maximum rarefaction obtained with tube No. 4, 1.1mm. diameter, was .66M.\* with an expenditure of 7,200c.c. of mercury at 15 mins. per 100c.c. = 18 hours. Going to the other extreme I next tried tube No. 1, 2.4mm. diameter, for high exhaustions. This gave, with 2,700c.c. of mercury, an exhaustion of .1m. in 2.5 hours. In the case of fall tubes Nos. 2 and 3 I have unfortunately not recorded sufficient data to get the quantity of mercury and time occupied as in the other cases, but practically I know that with these sized tubes the high exhaustions are most readily produced, and with them I have several times recorded .01m., and three or four times with my five tube pump, which contains three tubes in the centre, of 1.5mm. diameter, and one on each side of 1.8mm. I have measured exhaustions up to .000006mm. equal to .008M. Such exhaustions as these are, however, only produced under the most favourable circumstances.

\* M is now generally used to stand for a millionth of an atmosphere.



## THE MEASUREMENT OF THE VACUUM.

Moderate exhaustions, viz., to within 1mm. or so of the barometric height, are generally and most readily measured by means of a barometer gauge which forms a part of every air pump. Great care is, however, necessary in reading, in order to obtain trustworthy observations at pressures within two or three millimetres of the height of the barometer. A few remarks will therefore not be out of place on the methods used to obtain the necessary precision. The position of the barometer gauge in my arrangement is shown at *u* (Fig. 1), and to facilitate comparative measurements I place on the right of it a good barometer *v* and a glass rod *w* divided into millimetres measured from the extreme lower end, which is pointed in order that contact with the surface of the mercury may be seen by the point of the rod just meeting its reflection on the bright surface of the metal. As this operation had to be repeated for every reading taken, owing to the constant and considerable variation of the height of the mercury in the gauge reservoir, I devised the following method of obviating the necessity for constantly keeping the surface of the mercury bright, as well as stooping to see that the exact contact was obtained. A stout piece of platinum wire was sealed to the lower end of the measuring rod and filed to a sharp point, from which the millimetre divisions up the rod were measured. A thin wire was attached to this platinum point, which, after being twisted into a spiral to allow of the up and down motion of the rod, proceeded to a small galvanometer, which could be placed in any convenient position on the stand of the pump. Another wire from the mercury in the gauge reservoir passes through a small battery to the other terminal of the galvanometer.

Now it is only necessary before reading the height of the gauge to lower the rod till the first movement of the galvanometer needle is seen, which shows that the surface of the mercury has been reached. I must here mention that on visiting Prof. Barrett's laboratory at Dublin, I found that he had independently devised and was using the same arrangement for one of his barometers. In the absence of a cathetometer I still use a mirror placed vertically behind the top part of the barometer and gauge, in order that the reflected image may be used to prevent errors arising from parallax. The best method of reading the difference between the heights of barometer and gauge is to have a slide running smoothly on the measuring rod, carrying a piece of clear mica with fine lines, a millimetre apart, scratched upon it, which lines must move up and down at right angles to the gauge. Even if every precaution be taken, and the finest possible adjustments be used, it is still impracticable to obtain any approach to accuracy in the measurement of exhaustion above 1mm. or so by means of the barometer gauge. We therefore have recourse to the now well-known low-tension gauge of Professor McLeod, a slight modification of which I have used in my experiments. The modification consists merely in giving it a greater range and increasing the accuracy with which it can be calibrated. In Fig. 3 is shown an ordinary gauge, of which *V* is the volume, and *P* the pressure tube. In Fig. 4 it will be seen that on the top of *V* is blown a small volume tube *v* which is about 1in. long and 5mm. internal diameter, made of a piece of thermometer tubing, with white enamel back, the latter greatly assisting one to read quickly the position of the mercury in the tube. To avoid correction for capillary depression when using the small volume tube, I have placed a second pressure tube, made of the thermometer tubing, by its side. This second pressure tube communicates with

the large pressure tube *P* at *a* and *b*, so that mercury always rises in both at the same time.

In the calibration of the McLeod gauge it has always been difficult to obtain the total contents of the bulb and volume tube—that is, to the point where the gas is enclosed by the rising mercury passing the junction of the pressure tube at *c* (Fig. 3). Professor McLeod in his original paper, recommended pouring in mercury while the apparatus was held in an inverted position, till it runs down the pressure tube. It will readily be seen by reference to Fig. 3 that this method would only give approximate results. In calibrating several gauges I have overcome the difficulty by the introduction of what is technically called an improved joint *d* (Fig. 4), which is similar to the joint forming the air-trap of a barometer, the only difference being that the inner projecting part, *e*, is very short and has a smooth round opening of 3mm. or 4mm. diameter instead of being drawn to a fine tapering point. In this case the mercury, rising upwards, cuts the communication between the bulb and the pressure tube quite sharply at the smooth aperture *c*. It will also be seen that the introduction of this joint enables one to determine accurately the contents of the bulb and volume tube to the exact point where the rising mercury encloses the amount of residual gas to be compressed. The working of the gauge is in no way altered by these slight improvements. I might therefore refer those who are not familiar with its use to Prof. McLeod's original paper in the *Phil. Mag.* for 1874; but as this is not easily accessible to all, I will briefly describe the *modus operandi*. Let it be granted that the volumetric contents *v* to *e* is accurately known, and also that from *v* to division 60 in *V*. Although in working, the values of other divisions are taken in the volume tube, these two values are sufficient for the present description. On dividing the contents of the bulb and volume tube, viz., *v* to *e*, by the contents of the volume tube *v* to *V*, we have the ratio *R* existing between the two. Now let the whole apparatus be exhausted from *g* to *h*, and then allow mercury to rise from *h* upwards, as soon as it arrives at *e* the residual gas in *f* will be cut off from the pressure tubes *P*, and gradually compressed into the volume tube by the rising mercury filling up the bulb *f*, the increase of rise in the pressure tube *P* showing the pressure in millimetres *P*<sup>1</sup> of mercury necessary to accomplish this condensation, then

$$\frac{P^1}{R}$$

gives approximately the degree of exhaustion that existed in the bulb *f* before condensation. In order to find the exact original pressure, a slight correction is necessary, on account of the depression of *P*<sup>1</sup> by the tension of the residual gas in the pump with which the gauge is connected. To apply this correction the number

$$\frac{P^1}{R}$$

must be added to *P*<sup>1</sup>, and the result again divided by *R*. The difference between the number thus obtained and the approximate pressure is, however, so slight in the high exhaustions for which the McLeod gauge is generally used, that it comes considerably within the limits of experimental errors. For example, we will take a pressure of 113mm. approximate pressure, which is represented in the particular gauge I am now mostly using by  $\frac{11}{10}$ , that is, the residual gas requires 10mm. pressure to condense it to the division 60 of the volume tube. This approximate pressure added to the 10mm. in the pressure tube equals 10+113, which, when again

divided by the ratio, viz., 88.42, in this case equals .114—that is, a difference of 1 in the third place of decimals. This, perhaps, might be considered appreciable, but as most of the pressures measured by the McLeod gauge are far lower than this, I think it will suffice to show that the correction is scarcely necessary, especially as it involves considerable extra calculation.

#### CALIBRATION OF THE GAUGE.

The calibration of the McLeod gauge has always been a matter of some little difficulty, and the method I use being to the best of my belief new, and at the same time exceedingly simple and accurate, I will give it in detail, thinking it may be useful to those who, like myself, would not trust to a gauge unless they had personally verified its calibration. Fig. 5 represents the top part of an inverted volume tube *a*, the division on it to which we want to find, from the weight of the mercury with the meniscus in position *b*, what weight of mercury would fill the space to division *a*, when we are reading with the meniscus in the dotted position *c*. Make a wooden plunger to fit rather loosely in the volume tube to be graduated, and rub the end on a piece of fine glass paper till it is perfectly flat, then place mercury in the volume tube rather above the division which it is desired to calibrate, and slowly pass the plunger down to the division. All the excess of mercury will pass the plunger, and on withdrawing the latter this excess will be removed, the exact weight of mercury to the division selected being left in the tube. In this way I proceed to obtain the values of four or five different portions of the volume tube. To correct for the meniscus when reading with it in position (Fig. 4) I have only to find the value of the annular space *d* and add it to each of the values found by the above plunger method, since the meniscus will be of the same shape in all parts of the tube. The weight of mercury that would occupy the annular space *d* is found by taking accurately the weight of mercury to a certain division with the meniscus in position *b*, and subtracting this weight from that obtained by means of the plunger to the same division. The accuracy which may be obtained by the use of a plunger in calibration is shown by the following four weights obtained while calibrating a volume tube of 4mm. internal diameter to a certain division, 115.07 grains, 115.01 grains, 115.04 grains, 115.09 grains. In calibrating a McLeod gauge, it is advisable to select three or four of the most useful divisions and accurately determine the weight of mercury for each by the method described, calculate a table for various pressures on each of these divisions, and take all observations from one or other of them. The points in an ordinary gauge with the volume tube 60mm. long, which will serve for all pressures, are (1) the topmost division, (2) 5mm., (3) 20mm., (4) 60mm.

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MEETING, Wednesday, February 6, 1884.

### GASEOUS FUEL APPLIED TO THE HEATING OF GAS RETORTS.

BY CHAS. HUNT.

IF it were at all incumbent upon the writer of a technical paper to introduce his subject with a history of the art to which it relates that necessity does not

devolve upon me to-night; for at the last meeting of the Section our attention was largely occupied by the able survey of the rise and progress of illuminating gas with which Mr. George E. Davis appropriately ushered in his description of a proposed new departure in the economy of coal distillation. By so much, therefore, my task is a less formidable one; and if, of what remains, my subject be less generally interesting than those that have preceded it, I am not without hope that in practical importance it will compare not unfavourably with them, or indeed with any that may be brought under the notice of the Section.

Heating gas retorts by fuel in the gaseous form, as a substitute for its common application in the solid state, dates almost as far back as the introduction of the regenerative system by the late Sir William (then Dr.) Siemens, more than twenty years ago. The success that attended the latter in its applications to other industries led to its being applied experimentally in at least two gasworks in this country—namely, the Brick Lane station of the then Chartered Gas Company, and the Windsor Street works, at that time belonging to the late Birmingham Gaslight and Coke Company. In neither case, however, were the results such as to secure for it a more extended use, although, almost simultaneously, it obtained a firm and (as it proved) enduring footing at the works of the Paris Gas Company, where, with sundry modifications of detail, it has ever since been very largely employed. Here is a drawing showing substantially the arrangement which down to the year 1875 (when it was finally abandoned) was used in Birmingham for heating six beds of retorts. It is that of the familiar alternating system, with regenerators for both air and gas, worked by means of reversing valves, the producers (not shown upon the drawing) being of the ordinary kind, with sloping grate, placed at a convenient distance outside the retort-house, to which the gas was conveyed by the usual wrought-iron condensing tubes.

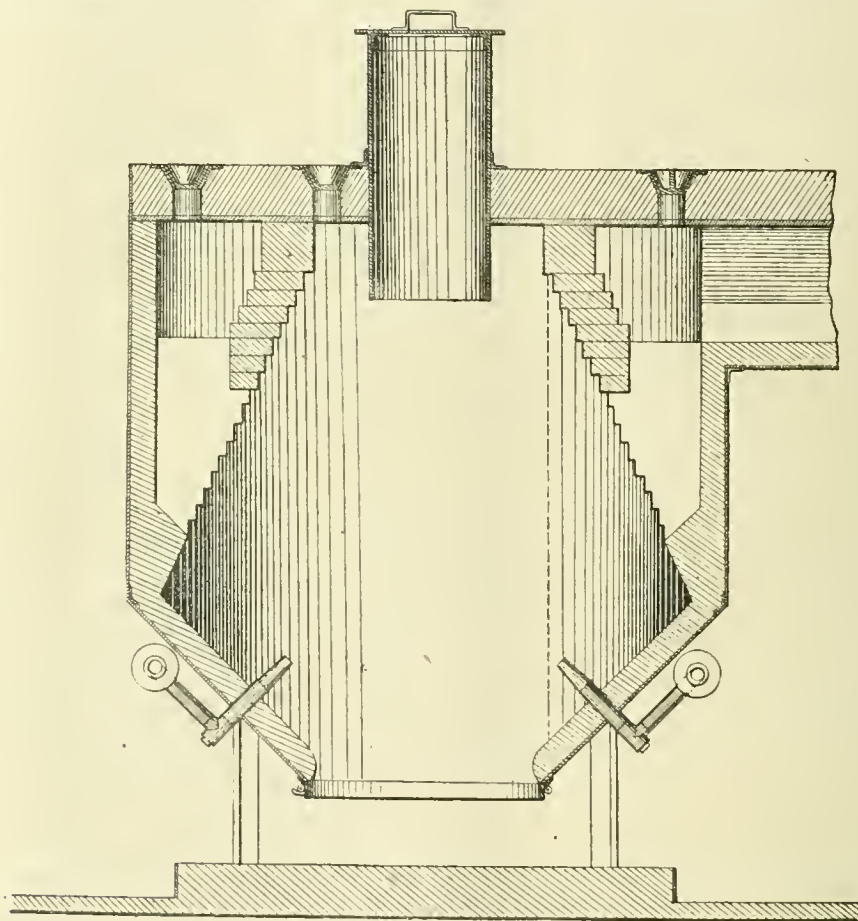
It is only natural to inquire how it came to pass that a system which, in its most important applications, has proved so advantageous, failed (in this country, at any rate) to displace, even in the smallest degree, the less scientific method of direct firing when applied to gas retorts. From personal experience I am able to reply that the small fuel economy and greater regularity of heating, which constituted the chief merits of the system, were too dearly purchased at the expense of simplicity, convenience, and certainty of action, the sacrifice of so much additional ground space as was required for the producers, and the employment of capital altogether disproportioned to the service performed. It may be only fair to assume that, under more favourable conditions, successive modifications might, as at the Paris Gasworks, have led to better results; but so far as the experiment (extending over several years) was pursued, the advantage remained with the more primitive system.

Other methods, however, for the attainment of the same object were, in the meantime, in process of development. It is very probable that the stimulus to further inquiry was furnished by the scarcity and consequent high prices of fuel that prevailed about ten years ago; and it may not be far wrong to conjecture that the clue to the direction which has since been followed by our German brethren with such assiduity and success was furnished by the Ponsard gas furnace—the first attempt, apparently, at continuous recuperation. I ought to qualify this observation by saying that it has special reference to recuperators built of fire-brick, because only within the last few days it has come to my knowledge that a patent was taken out as long ago as 1847, by



Richard Laming—a name well known to every student of the history of gas lighting—for a continuous recuperator made of iron tubes; and it is interesting to know that this patent describes a method of checking the primary air supply so as to generate carbonic oxide, and the introduction of a secondary air supply, upon the same lines as are now followed in the systems of regenerative firing recently introduced. It also appears that the invention was not at that time a new one, for subsequently Mr. Laming filed a disclaimer of that part of his patent, on the ground of want of novelty. It is, in fact, the adaptation of continuous recuperation that, by doing away with all necessity for valves and other compli-

were quoted, both of producer and chimney gases, showing that not only is it next to impossible, even under the most favourable conditions, to effect the entire conversion of fuel into carbonic oxide—a considerable percentage of carbonic acid being invariably present in producer gas—but also that there may be with the generator the same imperfect regulation of the air supply as with the ordinary furnace, concurrently with greatly superior economy. In addition to these, I am now enabled to refer to a series of seven analyses recently made by my colleague, Mr. Hack, at the Saltley works, which show an average of 5.2 per cent. of carbonic acid in the furnace gases, and of 9.3 per cent. of free oxygen in the chimney gases, the latter com-



SIEMENS GENERATOR (1881).

cations, has contributed more than anything else to render gas firing a practicable method for heating gas retorts. It would, of course, be idle to gainsay the advantages possessed by gaseous over solid fuel for many if not for most purposes, quite apart from the facility which it offers for economy in the recovery of waste heat; but the tendency is to overrate these, and to look upon gas firing as the principal if not the sole object to be accomplished, regardless, it would seem, of the fact that gasifying has to be resorted to, in connection with the regenerative system, as a means to the end, rather than as the end in view. In thus repeating an opinion expressed in the paper read by myself before the Midland Association of Gas Managers, in September, 1882,\* I am enabled to adduce further evidence in confirmation of its accuracy. In the course of that paper some analyses

paring with 9.7 per cent. in the case of an ordinary furnace, in which the consumption of fuel was nearly one-half as much again as in the regenerative furnaces. The latter, it is clear, owed little, if any, of their superiority to better regulation of the air supply.

The invariable presence of carbonic acid in producer gases—indicating to that extent complete combustion—points to the desirability of placing the generator in as close proximity as possible to the furnace, so as to avoid loss of heat in transmission. Nevertheless, the use of detached generators, in the large majority of its applications, goes far to prove that the economy of gas firing is independent of any moderate degree of imperfect gasification; nor is it conceivable that this can be due to any other cause than the recovery, more or less complete, of the waste heat. It is, however, objected by some that gasification alone is productive of the fullest economy, and

\* See *Journal of Gas Lighting*, vol. xl., p. 598.

ought consequently to be pursued as the object of primary importance.

With the view of determining this question, a Siemens producer (see diagram) was placed in a suitable position outside of a retort-house at the Windsor Street

#### ANALYSES OF GASES (SALTLEY FURNACES).

	Furnace Gases.					Waste Gases.		
	CO <sub>2</sub>	O	CO	H	N	CO <sub>2</sub>	O	CO
No. 1 .....	6.4	..	19.4	8.5	65.7	11.1	6.9	..
2 .....	5.9	..	20.4	6.5	67.2	10.0	8.9	..
3 .....	5.6	..	23.4	6.8	64.2	5.4	13.1	..
4 .....	6.0	..	21.3	8.6	61.1	6.5	10.7	..
5 .....	4.6	..	21.6	6.2	67.6	7.1	9.6	..
6 .....	3.6	..	22.0	8.4	66.0	9.8	6.8	..
7 .....	1.2	..	20.4	8.6	66.8	8.4	9.4	..
Averages .....	5.2	..	21.6	7.7	65.5	8.3	9.3	..

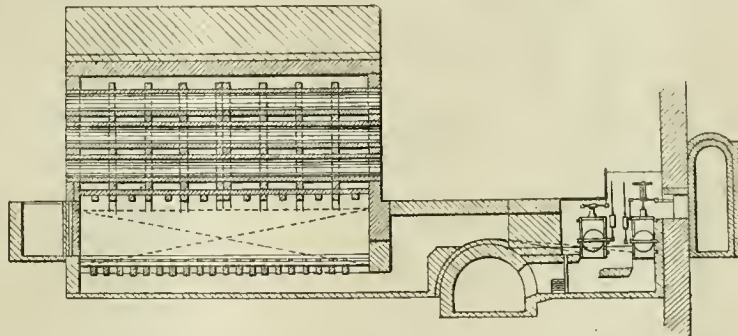
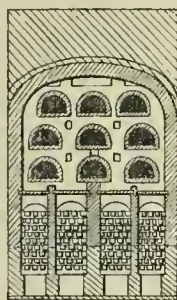
#### ORDINARY FURNACES.

10.3	0.6	0.3	5.4	83.4	8.7	9.7	..
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works, and the gas from it conveyed through an iron conduit lined with fire-brick, and provided with suitable branches, to two beds of retorts. No change was made in the construction of the latter, beyond closing up the space below the fire-bars, so as to

cation of the Wilson gas, Mr. Allhusen says: "We began by using cold air, which was a failure; then heating the air through metal pipes, which was an improvement; and now we have at last reached perfection by the adoption of a series of hot-air stoves, by means of which the air is forced into the combustion chambers at a temperature of about 1,000° Fahr."

Much attention has lately been attracted to the apparatus devised by Mr. Sutherland, and just referred to, for gasifying fuel under exhaust, so as to increase the generating capacity of the producer, and for extracting the tar and ammonia from the resultant gases. A word concerning it, in passing, may not, therefore, be considered out of place. It is hardly to be expected that gas manufacturers should regard with perfect equanimity the development of new sources for the production, in quantities sufficiently large to affect the market, of two of their most valuable by-products; but it may reasonably be doubted whether the effort of Mr. Sutherland is destined to have any appreciable effect in this direction. It is almost needless to observe, since the fact is now generally recognised, that tar which is produced under a condition of either partial or complete oxidation of the fuel is of a different character from that which is the result of distillation in closed vessels, conducted at moderately high temperatures, benzene being conspicuously absent and its place supplied by the comparatively



SIEMENS REGENERATOR FURNACE, AS FORMERLY APPLIED AT THE WINDSOR STREET GASWORKS, BIRMINGHAM

prevent ingress of air in that direction, and bring the necessary supply more completely under control. The gas was introduced at either side through openings made for the purpose in the furnace doors, the same means being employed for the admission of air, which was regulated as nearly as practicable to the requirements of combustion, as indicated by analyses made from time to time of the chimney gases. Periodical analyses were also made of the gases issuing from the producer, these showing the action of the latter to be satisfactory. The results obtained were a much lower heat in the retort beds than with the usual system of firing, and a great increase in the quantity of fuel used, the actual consumption being 6 cwt. of coke and slack per ton of coals carbonised, as against 3.53 cwt. of coke with direct firing, or a difference in favour of the latter of no less than 70 per cent. Almost simultaneously a similar experiment was in progress with Sutherland's apparatus, with analogous results. In both instances it was proved beyond question that, for heating gas retorts, gasification without recuperation was a step in the wrong direction. This experience has been curiously confirmed in connection with the application of gaseous fuel to another industry—that of salt-cake manufacture—by Mr. Allhusen, of the Newcastle Chemical Works, quoted by Mr. Wilson in the paper read by him at the first meeting of this section.\* Speaking of his appli-

valueless paraffin oil. It also appears probable that the same cause—viz., the admission of air, which thus operates so adversely—tends at the same time to limit the available production of ammonia, for this is yielded in much smaller quantity than usually obtains in the manufacture of illuminating gas, under conditions, as regards temperature, not unfavourable to its formation.\* The sum realisable per ton of coals by these products, tar and ammonia, has not yet, so far as I am aware, been determined upon sufficiently reliable data, but it cannot be considerable, while the cost of securing it, as represented by the loss of heat involved in the process, and the expenditure upon plant, is by no means insignificant. Speaking from actual experience, the latter amounts to at least five times as much per ton of coals as is found to be sufficient in the manufacture of illuminating gas for corresponding plant of greater efficiency; while, as regards the former, the cooling down of so large a volume of gases representing each ton of coals gasified, assuming this to be from 150,000 to 180,000 cubic feet, from a temperature of, say, 1,000° Fahr. to that which is necessary for the elimination of ammonia, about 60° Fahr., will be found upon calculation to represent something like 10 per cent. of the weight of the coal operated upon. Together, these two items make a formidable inroad into the saving to be effected by

\* See *Journal of Gas Lighting*, vol. xliii., page 231.

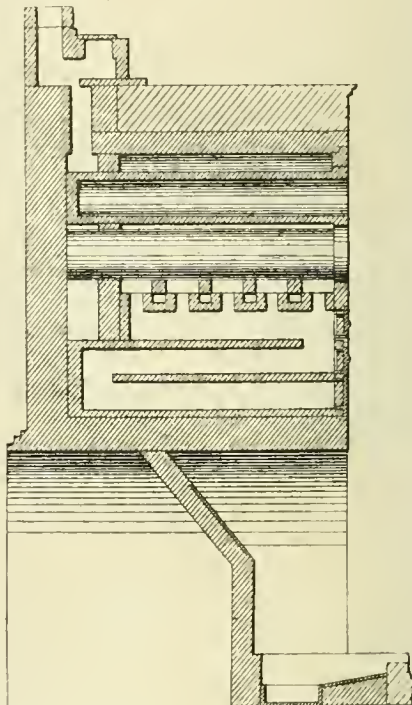
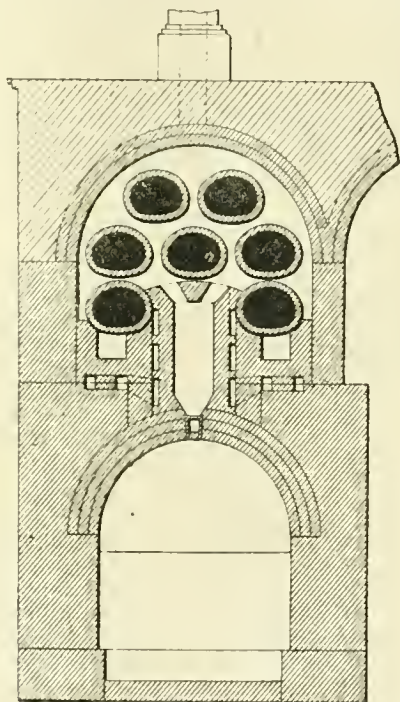
\* This was written previous to the publication of Mr. Watson Smith's paper, read before the Liverpool Section of the Society of Chemical Industry, December 5, 1883.



the extraction and sale of the by-products, to say nothing of the additional space required for the apparatus and the increased attention which it entails.

At first the new system of gas firing was applied to gas retorts with a very small amount of recuperation. It was, of course, necessary to introduce a secondary air supply, and the way to do this naturally suggested itself in the form of a longitudinal flue placed on each side of the furnace or gas flue, and extending almost to the back of the retort bed. These, being open at the front for the admission of air, fed combustion by means of openings placed at intervals throughout their entire length. When space permitted, two or more such flues were constructed lower down in the setting, air being admitted into the bottom one, and made to traverse backwards and forwards, always in the opposite direction to the waste gases, until it reached the top or distributing flue. Gradually more elaboration was introduced, until, in its best known

late Sir W. Siemens, being upon the same principle. Drs. Schilling and Bunte have adopted a grate generator, having fire-bars arranged very much after the manner of an ordinary furnace. The two last named are examples of detached generators. In addition to these three there is a fourth, which although regarded with less favour by the leading engineers of Germany, where it originated, has won approval in this country by its superior adaptability. This is known as the Klönne, a trial of which, two years ago, at the Adderley Street works, Birmingham, led to its adoption upon a larger scale at the Windsor Street and Saltley works, and was largely instrumental in causing its introduction in other places. At the present time it and the new Siemens system, already mentioned, may be said practically to divide the attention of English gas-makers on this subject, although other methods have been or are in course of being tried, and I shall have to refer to a recent modification of my own devising.



VALON'S GENERATOR FURNACE.

and most approved types, the modern regenerative furnace has assumed dimensions which, on first thought, appear to be out of all proportion to the work required of it, but to which the English gas-maker is fast becoming reconciled by the advantages it is found to possess.

Of the many systems that are now in use in Germany, it may be correct to describe as the principal ones the following—namely, the Liegel, the Dessau or Didier, and one devised by Drs. Schilling and Bunte, of Munich. In the first of these, which has what is termed a "slit" generator—i.e., a generator terminating in a long narrow slit for the admission of the primary air supply—may be recognised the prototype of the first generator furnace constructed in England, at the Old Kent Road Gasworks, by the then engineer (Mr. G. Livesey), and since improved by Mr. W. A. Valon, of Ramsgate. The second has a hearth generator, and this also is represented in this country; the producer in use at the Glasgow Gasworks, which originated with the

The Siemens system, as applied by Mr. W. Foulis, chief engineer to the Glasgow Corporation Gas Department, was fully described by him at the last meeting of the Gas Institute, held at Sheffield;\* and, briefly, it may be said to comprise a generator, or producer, placed outside the retort bed, formed by a wrought-iron casing lined with fire-brick, and having openings at the bottom, three or four in number, for promoting combustion. The fuel is deposited upon a hearth of fire-brick, there being no fire-bars, and at each opening a small jet of steam is applied, with the object of preventing the formation of clinker. A gas flue connects the producer with the setting, and is made continuous through the latter, the gas being thus made to meet the heated air issuing from the regenerator. This latter possesses one merit to perfection, that of extreme simplicity, the alternating gas and air flues being formed with ordinary fire-bricks and tiles. The producer is filled

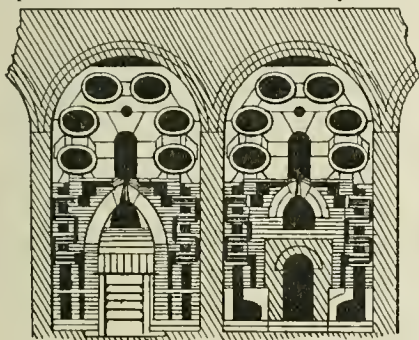
\* See *Journal of Gas Lighting*, vol. xli., p. 1139.

periodically with coke hot from the retorts, and requires no other attention than the removal of ashes and clinker from the hearth previous to being filled up with fresh fuel. Mr. Foulis thus describes the advantages of the system, which he has already applied to 900 retorts :—

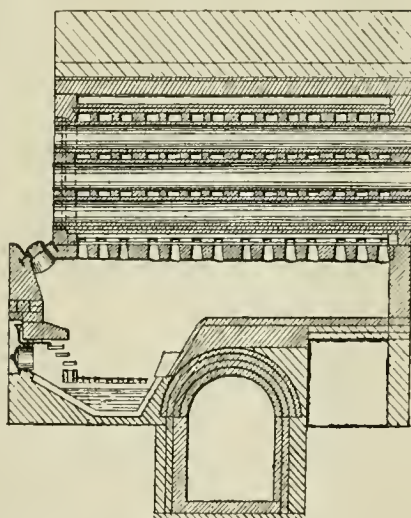
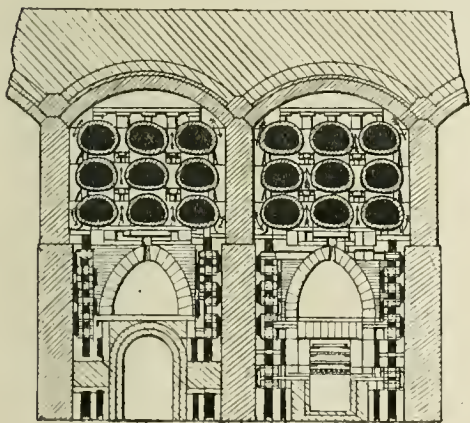
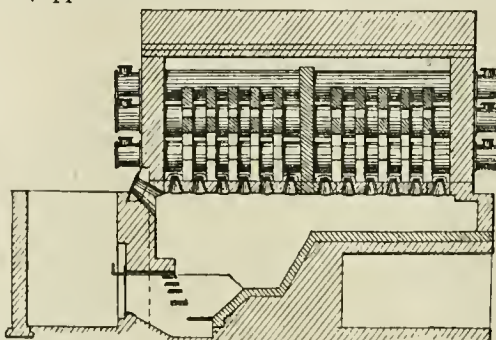
"A considerable saving in labour in firing; a decrease in the quantity of fuel used; an increase in the quantity of gas produced per mouthpiece; and greater durability of the retorts and settings. The saving in labour will be at once apparent. There is no firing, in the ordinary sense, and no clinkering. The saving in fuel we find to be considerable. As has already been pointed out the coke we use is very inferior, and consequently a much larger percentage is required to heat the retorts than when a better coke is employed. In ordinary furnace-heating we find it requires about 60 per cent. of the coke made to maintain the heats; with the regenerative furnace 33 per cent. is found to be sufficient, and we have a surplus of heat. The retorts are 13in. by 18in. and 9ft.

however opinions may differ as to the comparative merits of particular systems. As showing how very divergent these are may be cited that of Mr. W. A. Valon, of the Ramsgate Gasworks,\* who has applied a much less costly and pretentious arrangement, with, according to his view, equally good results. With Mr. Valon's efforts in this direction it is impossible not to sympathise, and it is certain that all who are interested in the development of regenerative firing will wish him further success; but, in the meantime, it must be admitted that his system has yet to be proved by comparison with others under different conditions than those existing at his own works.

The Klönne system has been applied very extensively at the Saltley works, and early last year was applied to 126 retorts at the Windsor Street works,



KLÖNNE'S REGENERATIVE FURNACE.



KLÖNNE'S REGENERATIVE FURNACE AS APPLIED AT WINDSOR STREET GASWORKS, BIRMINGHAM.

long, and they are charged every three hours. The production per retort—with ordinary furnace-heating, and with coals yielding barely 10,000 cubic feet per ton—is about 7,000 cubic feet in 24 hours. With the regenerative furnaces we get from 8,000 to 8,500 cubic feet in the same time. There is no dust or deposit of any kind on the outside of the retorts or in the setting, the brickwork after six months' work being as clean as when it was put up. The heats are very uniform, and are much less dependent on the firemen than with the old system. At present we have a producer to each setting of eight retorts. We have had, however, at work for some months a producer supplying the retorts on both sides of the bench. This plan is working well, and all the settings we are now erecting are built so that a similar arrangement can be carried out. In this case the regenerators, etc., remain as before; but the gas flue is carried right through from one side to the other. When one producer is used for both sides of the bench the fuel needed is only 25 per cent. Takirg the quality of the coke into consideration this is a remarkably low percentage."

This unequivocal testimony in favour of regenerative firing does not want for abundant confirmation,

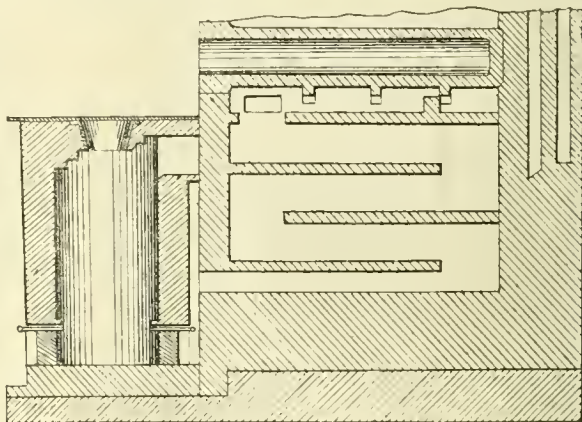
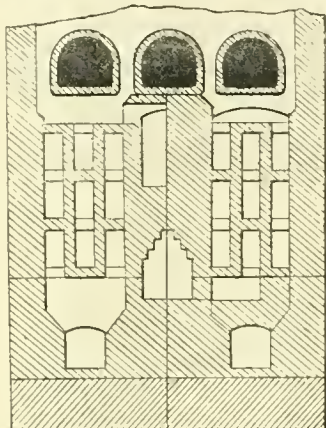
the plan of construction being essentially the same as had been previously followed at Adderley Street, with the addition of a provision for heating the primary air supply. On referring to the diagram it will be seen that the generator stands partly forward—about 2ft. 5in.—from the setting, being by this much a departure from the original conception of Mr. Klönne. This was arranged, at my suggestion, to facilitate the charging with hot coke direct from the retorts. The generator very closely resembles the first Siemens producer, having fire-bars in front, forming a succession of steps, immediately above the top one of which, and protected from the fire by a projecting row of fire-clay lumps, is placed

\* See *Journal of Gas Lighting*, vol. xli., pp. 269-70.

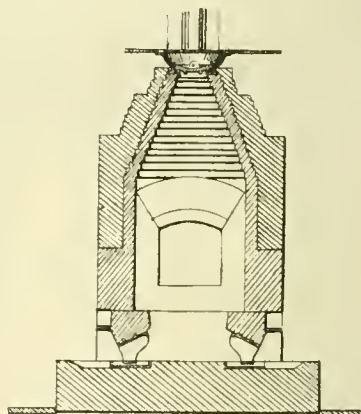
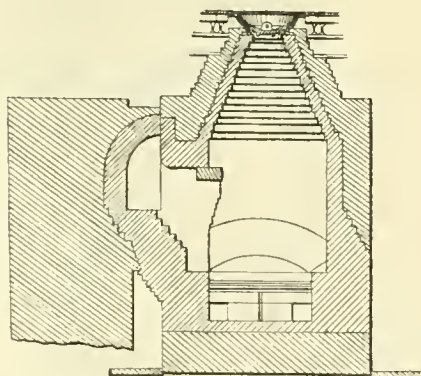


a shallow water pan. Water being continually fed to this pan overflows, falling from bar to bar into the ash-pan below. An air-tight or closely-fitting door, with adjustable ports, furnishes the means of controlling the primary air supply; and in addition side channels are formed, leading from the front to the under side of the fire-bars, by which means a certain amount of recuperation is effected. On issuing from the generator the combustible gases enter the setting through the openings at the top of the arch, and meet the heated air from the recuperators. These are formed of a series of special bricks, a specimen of which is on the table, having openings, in one direction for the downward current of waste gases, and in another (at right angles to the former) for the passage of the air,

Some inconvenience has been experienced with these settings, from the unequal distribution of this intense heat, by far the largest share falling to the bottom middle retort, which, in consequence, soon became a source of much embarrassment, and had to be removed. Where the system has been applied to settings having the same arrangement of retorts as is usual with direct firing, this difficulty has not been felt to anything like the same extent, the reason being that in these there is always a space corresponding with that occupied by the furnace, serving in a great measure to disperse the heat before it reaches the retorts. In other respects the system has fully answered expectations, the furnaces being easy to manage, the clinkering being done once in 24 hours (while at Saltley and elsewhere it is done only once in



SIEMENS GENERATOR, AS USED IN GLASGOW.



DESSAU OR DIDIER GENERATOR, AS USED IN DRESDEN.

which, introduced at the bottom of the setting, threads its way first to the right and then to the left, upwards, until it reaches the top highly heated. There is thus a constantly descending current of waste heat, divided only by a very thin partition of fire-brick—the thickness of material being only  $1\frac{1}{2}$  in.—from an equally constant ascending current of air, the conduction of heat being, under these conditions, almost as complete as is practically possible. The effect of this recuperation is to produce an exceedingly high temperature of combustion—so high, in fact, as to melt fire-bricks of the very best quality when brought within its immediate influence, and necessitating the greatest care in the disposal of the brickwork, so as to avoid undue exposure of any portion of it.

48 hours), and the fuel used has been found, by repeated measurement, not to exceed from 14 to 15 per cent. of the coke produced. This is only a little more than one-half the consumption by the old method, although it would not be fair to attribute the whole of it to regenerative firing, because the comparison in this case has been between settings of unequal capacity—namely, sevens by the old method and nines by the new—economy almost invariably being found to result from the heating of a larger number of retorts from the same source. Making, however, all due allowance for this, there still remains a very large margin—amounting probably to 33 per cent.—to be credited to the recovery of the waste heat. Nor do the advantages of regenerative firing end here. The temperature of

the retorts is maintained with greater regularity, there being no loss of heat in clinking, as in the old method, and is more under control, it being possible, by a slight alteration of the dampers, to vary it to almost any extent in the course of a very few hours, thus placing within easy reach the means of very considerably increasing their productiveness, without the necessity for a more than proportionate expenditure of fuel. It would be impossible to say this of the old method of firing, in which high heats, although by no means the invariable accompaniment, are seldom or never attained without a lavish expenditure of fuel. The reason for this is to be found in the fact that the waste heat necessarily leaves the setting at or near to the temperature of the last retort, so that the more this is augmented the greater, by the old method, must be the loss, whereas by the use of regenerators of sufficient capacity a very large proportion is restored to the furnace. How great is the margin for saving in this direction may be learned, not alone from actual experience but also from calculation. A French writer upon this subject (M. Melon), in a paper\* read by him in 1881 before the Société Technique de l'Industrie du Gaz en France, thus classifies the sources of loss of heat from a retort setting:—

- (a) Loss by the chimney.
- (b) Loss by radiation from the front.
- (c) Loss by radiation from the foundations, etc.
- (d) Loss by the escape of distilled gases.
- (e) Loss in the furnace, by the formation of water and clinker.
- (f) Sundry losses, due to the opening of the furnace for clinking, the opening of the retorts during drawing and charging, etc.

And his summary is as follows:—

	Per cent.
Loss by the chimney .....	60.0
Loss by the products .....	17.0
Loss by the casing .....	2.5
Loss by the radiation .....	1.5
Loss by the furnaces .....	2.5
Sundry losses (estimated) .....	5.5

89.0

Leaving 11 per cent. as the proportion actually realised in carbonisation.

These estimates are based upon the assumption that the gases leave the setting at a temperature of about 2,200° Fahr., and are therefore evidently intended to apply to the employment of high heats. Where, as is most usual with us, the retorts are worked at six-hour charges, the temperature of the escaping gases does not probably exceed from 1,600° to 1,800° Fahr., and the loss by the chimney is consequently less. The figures, however, afford a sufficiently clear indication of the enormous loss simply inevitable under the old system, more than one-half of which is already in course of being arrested by the introduction of continuous recuperation. Whether more than this is possible remains to be seen, although a further reduction appears highly probable, from the fact that the escaping gases still leave the settings at a temperature varying from 600° to 900° Fahr., which is much more than sufficient for the maintenance of the necessary chimney draught. The cost of the regenerative portion alone of these settings of nines amounted to about £7 per mouthpiece, which is more than had been anticipated, and greater, in my opinion, than should be needful for the purpose. Being doubtful also of the effect of the arrangement for distributing the heat (which subsequent experience proved to be fully justified), I had, previous to the completion of the settings, devised a modification, constituting an entirely new recuperator, although proceeding upon the same lines, with a view of meeting, at any rate in some measure, both these objections. I hoped to have been able to lay before

the members the details of this arrangement, but as the latter may become the subject of a patent, I am advised that it will not be desirable to do so. I will therefore content myself with saying that in its effect, local heating, if not entirely avoided, is, at all events, less pronounced than in the Klönne setting; the consumption of fuel is no greater, being 1½ per cent. of the coke produced; while in the cost of construction there is a saving in material and labour of rather more than £20 per bed. Other improvements are now contemplated, by which it is confidently anticipated that further economy in cost of construction will be realised, involving a still more complete departure from the original system. The generator has also, in my arrangement, undergone considerable alteration, being brought out farther from the setting, and built up square at the back, so as to admit of its being filled up more easily. The charging-door, also, is placed in a horizontal position, this being found more convenient than the angular one imposed by Mr. Klönne. In this I find I have been anticipated by MM. R. Radot and A. Lencauchez, whose arrangement, as used at the Bordeaux Gasworks, will be found illustrated in the current volume of the Transactions of the Société Technique de l'Industrie du Gaz en France.

In the first application of any new system, difficulties have almost inevitably to be encountered. Whoever has not met with them with regenerative firing has something yet to learn. Speaking for myself, they have led me to take a keener interest in the subject than perhaps under easier conditions I should have found convenient, and have forced me to try conclusions that otherwise I might have avoided. It has been shown me, for example, that it is one thing to produce an exceedingly high initial temperature and another to apply it without inconvenience throughout the setting. The former is comparatively easy, there being many more ways than one of constructing an efficient, continuous recuperator; but the latter, while more difficult, is an absolute necessity for comfortable and economical working. The saving of a few pounds of fuel may be dearly purchased at the cost of a superabundance of stopped ascension-pipes and pitch in the hydraulic mains, to say nothing of injury to the brickwork of the settings. Local heating is a fruitful source of all these evils, and requires to be guarded against with the utmost care. For its avoidance a sufficient space should in all cases be provided to admit of the dispersion of the heat before it reaches the first retorts; and it will be found that where the centre flues have a width between the retorts of not more than eight inches, or where there is no space corresponding with the furnace of an ordinary setting, the plan adopted by Mr. Foulis—of a two-chambered setting, in which the heat is made to rise through all the flues in the front, and descend through all the flues in the back or central chamber—is to be preferred to the more usual one, by which the heat passes up the centre of the setting, over the top retorts, and down by the outer flues. Great evenness of heating is in this way insured; and for the rest, those who incline to working at high heats and short charges must depend, for the avoidance of stopped pipes, upon a light water-seal secured by the removal of the tar from the hydraulic main as rapidly as it is deposited. Another point of importance for the prevention of injury to the brickwork or retorts from the excessive temperature at the point of combustion is the formation, in its immediate locality, of a continuous open chamber, in which the first intensity may spend itself, as it were, before coming into contact with the brickwork. Attention to these matters of detail will be fully repaid; and a little skill brought to bear in the

\* See *Journal of Gas Lighting*, vol. xxxviii., p. 423.



construction of the settings will be found more to the purpose than a search, prosecuted to the ends of the earth, after the best fire-resisting material.

In the construction of a recuperator, certain conditions are necessary to be observed. These are the employment of the thinnest possible material consistent with strength and durability, and the securing of long contact, both for air and waste heat. When at Munich a few years ago, I remarked to Dr. Schilling, who was then experimenting with his regenerative furnace, upon the labyrinth of passages through which the air was made to travel by his arrangement, observing that it appeared unnecessary. He replied that it was a mistake to suppose that air could be raised to a high temperature otherwise than by long contact with a heated surface; and so it has proved in our experience. Not only does the air, in its passage upward take the shortest way, but also the waste heat in its passage downwards, if the draught be at all keen; and therefore the recuperator should be constructed with a view to the longest practicable retention in both cases. Since, also, the absorption of heat by the air is dependent upon conduction through the material employed, it follows that the thinner the medium the more rapid must be the effect. When the recuperator is constructed of material of the average thickness—say  $2\frac{1}{2}$  or 3 in.—very much greater space must be needed to secure the same degree of efficiency.

As to which is the best form of producer, opinions differ, and will probably continue to differ. When there is sufficient depth available to admit of the clinkering being done from below, the slit generator offers the advantage of simplicity; but whether it is equally suitable for all kinds of gas coke is doubtful, since experience seems divided upon the point. Nor does it appear at all possible, unless the setting be of considerable width, to apply it in connection with the most approved forms of recuperators; while the inevitable radiation from the walls and arch of the tunnel affording access to the under side of the generator must be a source of considerable loss. Otherwise, as a matter of economy no less than of convenience, there can be little doubt that the generator is in its proper place when forming a part of the construction of the setting, instead of, as in some systems, detached from it. This conclusion is inevitable to those who, like myself, have had an opportunity of comparing the two systems of construction side by side. Radiation from the enclosed generator may be almost entirely prevented, and is so inappreciable as to be almost imperceptible; whereas, on the other hand, from the detached generator—the Siemens, for example—it is most painfully evident. In cost of attendance, also, according to my experience, the latter compares unfavourably with the enclosed generator.

Depth of fuel has generally been considered essential to gasification, although the analyses published by Mr. Foulis show that conversion to carbonic oxide is, if anything, more complete when the fire has burnt low, than after it has been freshly charged. My own observations confirm this, and lead me to say that in all respects the dimensions of a generator should be determined solely from considerations of general convenience. A shallow fire requires replenishing more often than a deep one, and that is practically all the difference between the two; while a fire having a large area of grate surface will last longer without clinkering than one with a lesser area. Having tried several kinds, I have come to the conclusion that the advantage of a long interval without clinkering is more apparent than real; and that greater regularity of heating is secured by a comparatively narrow grate, clinkered once every twelve hours, than by a very wide one which is allowed to run twice or even

four times as long without being clinkered. Local circumstances, however, must largely decide both the dimensions and form of the generator to be employed; but, in any case, it is desirable to effect control of the air supply by means of a tolerably close-fitting door, furnished with adjustable ports similar to the furnace doors of Cornish or Lancashire boilers.

#### ANALYSES OF GASES, MADE BY DR. WALLACE, AT GLASGOW.

	1 p.m.	3-30 p.m.	5-40 p.m.
Combustible gases—	32.7	31.7	37.6
CO and H ..			
Non-combustible gases—			
CO <sub>2</sub> ..	7.2	5.3	3.4
N ..	60.1	60.0	59.0
	100.0	100.0	100.0

Although for the most part my observations have been necessarily addressed to a somewhat limited audience, composed of my professional brethren, I am anxious not to conclude without making some attempt at showing that the subject, far from being of a special nature, interesting only to gas makers, is really susceptible of very much wider treatment. It has been explained that, in addition to other advantages derived from continuous recuperation as applied to gas-retorts, not the least considerable is the saving in fuel, varying, according to the efficiency of the system, from 20 to upwards of 40 per cent. Speaking roundly, this means that for every ton of coals used in the production of illuminating gas, an additional hundredweight of coke is rendered available for other purposes. What this may amount to in a comparatively short space of time it is not difficult to estimate. In Birmingham alone it is not improbable that within the next few years the annual output of coke will, from this source, be increased by from 12,000 to 15,000 tons; while the yearly consumption of coals for gas-making purposes throughout the kingdom suggests a possible addition to our fuel resources of from 300,000 to 400,000 tons of coke per annum. This ought to be nothing short of a national benefit, although directly affecting only one particular class—namely, the consumers of gas; who, whether supplied by companies or by corporation, derive, with few exceptions, the full advantage of all reductions in the cost of manufacture. It may not, however, be at all safe to assume a money advantage corresponding with this enormous saving—for enormous it is—since even at the present time, it is only too evident that, notwithstanding its smokeless character and consequent adaptability to many requirements, gas coke does not command that full appreciation which its merits certainly deserve. This observation is much less applicable to Birmingham and its vicinity than to other localities that shall be nameless, whence relief from heavy stocks is habitually sought by appeals to distant markets; nevertheless, it is apparent that even here, in this great centre of manufacture, with its huge importations of fuel, gas coke occupies a far less prominent position than is its due. Why this should be so it is not within the scope of this paper to attempt to determine; but as a kind of natural sequence to the subject we have been considering it falls to me to direct attention to a very wide field for the employment of gas coke, hitherto all but absolutely unexplored.

A separate service of heating gas has long been a favourite idea of men in the highest walks of science; and at 1s. per 1,000 cubic feet, or thereabouts, has almost come to be looked upon as one of the greatest boons the future can possibly have in store for us. It is, however, generally forgotten that gas at such a price, or even at 6d. per 1,000 cubic feet, might still be relatively dear, in consequence of its low calorific value; the fact being that the very best heating gas that has



been proposed for distribution has offered no more favourable comparison with ordinary illuminating gas than as 3 or perhaps  $2\frac{1}{2}$  to 1. Its use could not, therefore, pay as against the latter at the price charged (say) in Birmingham or other large towns. But while the argument in its favour is thus altogether inconclusive, it is not equally clear that the instinct which has prompted the demand is erroneous or unsound. On the contrary, there is evidence enough to show that in every manufacturing district, and possibly for domestic purposes also, a really low-priced heating gas would be largely patronised. The question is how it may be best obtained, by an extension of the system of illuminating gas, or by an entirely new departure? As regards the former, the gradual diminution in the cost of manufacture is tending to bring illuminating gas into very general request for heating purposes; and this it has been proposed to further encourage by the rather doubtful expedient of differential rates. At the same time it has been repeatedly shown that the establishment of separate fuel-works would stand no chance of success in competition with the existing gas system, which, at a comparatively small cost, is susceptible of development to any reasonable extent; and therefore in this direction it has become the custom to seek for a solution of the problem.

Quite recently an important contribution has been made to the subject by the *Journal of Gas Lighting*, in commenting upon a letter from a correspondent who, writing under the *nom de plume* of "Progress," advocates the conversion, at gasworks, of coke into water gas, for distribution at a cheap rate. It has been remarked in the editorial columns that the consummation wished for by "Progress" may be much nearer than he imagines, inasmuch as the rapid extension of the generator system is furnishing, to those who may choose to apply them, the means of gasifying all surplus coke at a minimum of cost, and without the necessity of any material alteration or interference with the present system. I believe this to be one of the most reasonable suggestions on the subject that has yet been offered; and if ever a separate supply of fuel gas becomes a reality, it should be by such a method. Having devoted to generator firing a considerable amount of attention, I am disposed to say further that, given a real demand for a rough heating gas, there are no insurmountable difficulties in the way of supplying it in the manner proposed, and at a very moderate cost, as compared with what would be incurred by the establishment of separate works. In a stage house the requisite space is already provided. The saving of heat by filling the generators direct from the retorts would largely compensate for the loss involved in gasifying and in the subsequent cooling of the gases; while there would be an additional set-off in the almost entire absence of manual labour and waste in handling the coke. There seems to be really no reason why, if desired, the whole of the coke should not leave the retort-house in the form of combustible gases, ready, after cooling, for distribution in the ordinary way. Whether, on a balance of considerations, this would be economically desirable, is too wide a question to enter upon at the present moment; but it may be at once pointed out that not a little of the commercial success of such an undertaking must depend upon the means available for the application of the gas. Experience has shown that for many purposes even the heating power of illuminating gas is insufficient; and we also know that the application of carbonic oxide, or water gas, is by no means uniformly successful when combustion is promoted by cold air. From this it may be fairly inferred that the difficulties to be met with in the introduction of a purely heating gas are by no means inconsiderable, although

perhaps not so formidable as to absolutely forbid the enterprise. In the meantime, however, why should not heat users avail themselves of the means which are already at their command for securing all the advantages that can possibly be derived from gaseous fuel when a high temperature is necessary.

The earliest attempts at recuperation were by means of what is now called the primary air supply; and only when this was found to be unworkable—doubtless owing, in a great measure, to the overheating of the fire-bars—does it appear that recourse was had to conversion of the fuel into combustible gases. This was the great step that led to success, although accompanied by the separation of the producer from the furnace, and the consequent cooling down of the gases in their passage from the one to the other. The gases, however, thus became an additional vehicle for the recovery of waste heat, rendering the familiar two-chambered recuperator an inseparable part of the system. Of the great original conception of the brothers Siemens it would be superfluous to speak in terms of even the highest admiration, because the immense benefits it has conferred upon the manufacturing industries of this and other countries are universally acknowledged. But in the application of a principle there can be no such thing as finality, and it is only natural that efforts should be continually directed to simplifying the means by which the desired effect may be secured. Notably the periodical clogging up of the different parts of the apparatus with carbonaceous matter, apparently inseparable from the use of raw coal, has given exercise to much ingenuity. It gave the first impulse to the invention of Mr. Sutherland, and not improbably to others also, which have the same object in view, although proceeding upon entirely different lines.

But must it not be said that recuperation is still beyond the reach of all but the large capitalist, or resorted to only for special purposes, requiring the maintenance of an exceptionally high temperature irrespective of other considerations? At all events, it is certain that its use is confined to a comparatively few industries, notwithstanding that the scope for its employment is practically limitless. On the other hand, may it not be suggested, with very good reason, that in one or other of the systems of continuous recuperation now in course of introduction will be found a thoroughly reliable, comparatively inexpensive, and most manageable substitute for the alternating system, without the loss of any of its recognized advantages? It may be objected to this that I am advocating, in the interests of one particular manufacture—that of illuminating gas—the use of gas coke in place of raw coal; but I do so in the general interest no less than in the interest of the consumer of the fuel, who would be advantaged by the change, even though he might have to pay a somewhat higher price for the substitute. Already continuous recuperation is, I am informed, in course of introduction in connection with the Simon-Carves coke oven and Hislop's process for the recovery of lime; and it is to be hoped that manufacturers in this district will not be behindhand in showing what may be done in this direction by very simple means.

In the application of the original Siemens system of regenerative firing at Windsor Street, the fuel expenditure (slack being used) was 3·4 cwt. per ton of coal carbonised, against 4 cwt. of the same fuel required with direct firing. With coke as the fuel, the consumption by the latter method has averaged rather more than  $3\frac{1}{2}$  cwt. per ton of coal carbonised; whereas by gas firing with continuous recuperation it has been proved possible to effect the same work with 2 cwt. Should not this be an incentive to other heat users to do likewise?



## DISCUSSION.

Mr. G. E. STEVENSON : Mr. Chairman, I think, sir, it is very kind of you to invite a stranger, who is not a member of this Society, to take part in a discussion here, and I thank you for doing me the honour, and us generally, as gas engineers, of inviting us to speak here to-night. The subject of regenerating furnaces is one which has interested me, I may say, for several years, and one to which I have devoted considerable attention, though it has not been my lot to make practical experiments with the principle to any great extent. I may say my attention was directed to this subject, in the first place, by seeing in Germany the earlier attempts that were made by engineers to apply regenerative furnaces for the heating of gas retorts. It may not, perhaps, be known to all present here that German gas manufacturers have always been in the habit of heating their retorts to a much higher temperature than we in England, their custom being to make their retorts yield a larger quantity of gas in 24 hours than we have been in the habit of doing. One reason, perhaps, is the illuminating power is not so high as that which is required by the legislation with regard to our own gas supply, and therefore they were enabled to get a greater yield of gas out of the coal. I may say that when I first returned from Germany I began to mention this subject to my friends and colleagues, and I think I may take credit myself of contributing towards creating an interest in this subject. In Birmingham it was my privilege to read a paper introducing drawings of the Liegel furnaces used at Frankfort and elsewhere before the Association of Midland Gas Engineers, and from that time Mr. Hunt took the subject up with success. We have to-day seen the furnaces at Windsor Street and Saltley, and the style he has changed his furnaces to is a more economical one; and as Mr. Hunt has told us, there are now thousands of tons of coke saved in a year which were formerly used in heating retorts. There are one or two points in Mr. Hunt's paper to which I should like to refer. I was rather surprised to see in the table of analyses taken from the Saltley furnaces, that so large a portion of oxygen was present in the waste gases; and as the amount is about the same as that present in ordinary furnaces, it appears to lead Mr. Hunt to conclude that apart from regeneration there is no advantage in gas-heating furnaces. I cannot say that I come to that conclusion. The reason that large quantities of oxygen are found in the exit gases may be due to the imperfect air-tightness of the regenerating flues; and if that be so the air that produces the oxygen has entered the setting of retorts after the heated gases have done their work and has no influence on combustion, so as to lower the temperature of the retorts. My opinion about the matter is that gas-heating furnaces are decidedly superior to ordinary furnaces. I think that regeneration is a great additional saving, but where it becomes very elaborate in construction it is a question in the minds of all whether the construction pays for the saving effected. The matter has hardly yet been worked out. There was also a question about the dispersion of the heat in the settings. Mr. Hunt stated that although it was easy to create an intense heat it was rather difficult to obtain a proper dispersion of that heat, so that the whole of the retorts and brickwork should be heated to a uniform temperature. The principle to be aimed at is that combustion of the carbonic oxide gas should take place slowly, and not all at once. In the earlier forms of furnaces, such as that used by Mr. Valon, the air supply is delivered into the furnaces in a large volume, and in that case a

very intense combustion takes place. The heated gases, after combustion, pass through the retort setting in the usual way, and a very intense initial heat is created, which decreases as the gases pass in and come in contact with the retorts. If the air is mixed with the carbonic oxide in small quantities, the combustion takes place gradually throughout the whole length of the setting, and it is then possible to obtain a slower combustion, so that by the time the waste gases reach the exit flue, and are leaving the retorts, combustion ends. Of course it is difficult to heat air to a high temperature without keeping it a very long time in contact with the heated surfaces of the material by which it is to be heated. Air, being a bad conductor of heat, does not take up the heat so rapidly as water vapour, for instance, would do. In regard to the slit generator, this is one of the most economical descriptions of generator, but it is not suitable for all kinds of coke. With the coke from some kinds of coal the slag can be run out without difficulty, but with other kinds it is not possible to get rid of the slag or clinker in that manner. The use of gaseous fuel is a question of great interest, not only to gas engineers but in regard to a great many industries. We all know the waste of fuel in many ways is enormous, and it is only by controlling the combustion of fuel that we can prevent this.

Mr. HEAD, as manager for the representatives of the late Sir William Siemens, was glad to respond to the call made upon him by the chairman to join in the discussion. He would first congratulate the meeting upon the circumstance that they were not present to discuss the question of the adoption of regenerative gas furnaces, but to what form they should give the preference. That was a great step in advance, showing that the late Sir William Siemens's work had taken deep root in the minds of gas engineers, and had Sir William Siemens been spared to them, he (the speaker) was sure it would have given him great pleasure to have attended that meeting. The author had commenced his paper by a reference to an application of the Siemens furnace made at Windsor Street station twenty years since. That application was made according to the early form of the regenerative gas furnace, with reversing valves, a form which had received an extensive application at the Paris Gasworks, but it was not considered a success at Birmingham. The reason of that non-success, he recollected, was due to leakage of water into the regenerators, which would have been avoided had the furnaces been constructed with stage floors. The author remarked that there was no finality in the application of scientific principles, a statement which would have been heartily confirmed by the late Sir William Siemens, whose whole life was devoted to improvements in various engineering and scientific appliances, to none of which he gave more attention than to the regenerative gas furnace. He (the speaker) for twenty-three and a half years had had the privilege of assisting him in his civil engineering work, consisting mainly in the development and application of the regenerative gas furnace. This work would now be directed by Mr. F. Siemens, who during the whole time had been associated with his brother, the late Sir William Siemens, in the application of different forms of regenerative gas furnaces to meet the various requirements of manufacturers. The author seemed to think that the Siemens furnace, as applied to the heating of gas retorts, was necessarily coupled with the use of a gas producer outside the setting, and he (the speaker) wished to remove that idea as being an erroneous impression. As the late Sir William Siemens pointed out at the meeting of the Gas Institute at Sheffield last June, the selection of the form of gas producer, whether outside or inside

the setting must depend upon local circumstances, and as a matter of fact he could add that the Siemens furnaces at Halifax and Denton were provided with gas producers inside the setting, this form having also been adopted at Saint Mandé twenty years since with some of the earliest applications made by the Paris Gas Company. The outside gas producer was adopted for certain kinds of fuel, and in Scotland, where the coke was of a hard, slaty nature, it was found easy to work, and the analyses which the author furnished of the gas made in that and other kinds of producers showed quite in its favour, the gas from the former containing about 37 per cent. of combustible elements against 29 per cent. from the latter, thus compensating for the loss of heat by radiation, which was the objection raised against it. The gas producer shown in the diagram in connection with Herr Klönne's type of regenerative gas furnace, as mentioned by the author, was the mere reproduction of one of the Siemens early forms. That form, however, presented certain inconveniences in working which caused the late Sir William Siemens to modify it, shortly before his death, by the substitution of a solid support for the horizontal grate-bars, also by the suppression of the steps in front, in lieu of which an opening was provided at the level of the solid support for stoking. Clinkers would gather on the solid support, and could easily be pushed towards an open space at back, which served for removing them and also for the admission of air to the gas producer. The outside gas producer shown in connection with the Siemens furnaces at Glasgow had been likened to the Dessau or Didier form; but he (the speaker) disclaimed the resemblance, for in the latter case the outlet for gas was placed near the hearth or bottom, so that the outflowing gas would consist mostly of carbonic acid, whereas in the other case the outlet for gas was at the top of the column of fuel, and the analyses already referred to proved the gas to be of superior quality. It would thus readily be seen that this difference between the two apparatus, and there were others, was an all-important distinction. Touching the richness of gas made in producers, the speaker agreed with the author that the greater proportion of carbonic oxide and the smaller proportion of carbonic acid depended upon intensity of heat inside the producer, but this must be coupled with a slow, or at any rate a moderate, rate of combustion. He (the speaker) had personally carried out some experiments for the late Sir William Siemens which he considered settled that point, and dealing with Staffordshire coal in the improved type of Siemens producer he had obtained a gas containing only 2 per cent. carbonic acid with 0.2 per cent. of free oxygen. Ordinary workmen could not, however, be trusted to work under the conditions necessary for obtaining these results, as yet, although he found that in ordinary practice they obtained from Staffordshire coal a gas containing 48.6 per cent. of combustible elements, with only 3 per cent. carbonic acid and 0.8 per cent. oxygen. As mentioned by the author, the arrangement of gas and air jets in the combustion chamber, shown in the diagram of the Glasgow furnace, originated with Mr. Foulis, the well-known gas engineer of the Glasgow Corporation Works, and, with his permission, the late Sir William Siemens adopted it for his gas retort furnaces. To the zig-zag regenerator flues shown in the diagram, with which the late Sir William Siemens had commenced, he afterwards added baffle bricks. By that addition their efficiency had been insured under all conditions of working. He (the speaker) claimed the Siemens furnace to be as efficient as any other regenerative gas furnace, and much cheaper to build, bricks of ordinary size being used throughout, instead of costly and cumbrous special bricks like the one

shown upon the table. The results obtained at gas-works varied a little, but he had furnaces working with only 10lb. of coke to the 100lb. of coal carbonised, which would, he thought, be considered a satisfactory result.

Mr. HEAD asked to be allowed to say a few words touching a remark made by Mr. Paterson as to stoppage of ascension pipes. He (Mr. Head) was satisfied, from experience recently acquired, that stoppages were not due to the intensity of the heat of regenerative gas furnaces but to its misapplication. At Rochdale, a Siemens furnace had been set to work, since about four weeks, without any stoppage whatever of the ascension pipes occurring.

Mr. HACK: I have given considerable attention to this question, and am of opinion that the matter is no longer in an experimental stage. The principle is now well known to most gas engineers. My attention was first called to it on a visit to Paris, where the system was in operation at Clichy. I afterwards became more acquainted with it at our Adderley Street works; and from reading subsequent papers on the subject I determined to give it a fair trial. The theory seemed correct enough, and I started with the object of (1) realising a large saving per cent. in fuel, (2) saving in labour, and (3) increased capacity of the works by a larger yield per mouthpiece. I commenced first with a few of Klönne's settings, and after I added a modification of the Clichy furnace. I have also adopted the system in two or three ways devised by myself. With regard to the saving in labour anticipated, I am sorry to say that so far it has not been realised. The saving in firemen's wages has been more than nullified by the large number of men required to unstop the ascension pipes, which the increased heat caused. This drawback seems to me almost inseparable from carbonising at high heats. With regard to the fuel account, all that I anticipated has been borne out. In some of the furnaces on the Klönne system we have used but 10lb. per 100lb. of coal carbonised with similar settings (nine retorts in a setting). On the ordinary plan we were using 22 per cent. With regard to the Clichy plan, that is found not so economical as against the 22 per cent. formerly required. We are now using in these regenerators 17 per cent. In the remainder of the new settings we have used from 13 to 14 per cent., all of them showing a considerable saving in fuel, and I think that every gas engineer should no longer hesitate in adopting the new principle. In doing so at Saltley, I have had to contend with some rather difficult circumstances, having to alter benches that were originally constructed for ordinary furnaces; but even with these disadvantages I am so thoroughly satisfied that the principle is the correct one, that I hope to see it applied shortly to nearly every bench in my works.

Mr. SMITH CASSON: With the chairman's permission, I should like to say a few words on gas furnaces, having had several years' experience with them, the result being that I have come to the conclusion that for most manufacturing purposes regenerators, which, as is well known, are most costly in construction and maintenance, are altogether unnecessary, provided the air is heated in a proper manner, which is the case at the Earl of Dudley's Round Oak Iron-works, where the bulk of the iron is puddled, ball-furnaced, and reheated on my system. As showing the heat that we get, I may state that we frequently have occasion to heat 25cwt. "piles" or "blooms," which we easily do in an hour's time, reheating the same in 20 minutes. We have also melted wrought-iron scrap in crucibles in about 30 minutes, which proves the intensity of heat that we obtain. The gas is made in the ordinary producer, and the air heated in a somewhat similar manner to the Klönne system,



but if anything simpler, the furnace-man having perfect and instantaneous control over the gas and air, both being under pressure. I quite agree with Mr. Head in his remarks about the Tusard producer, as gas is drawn off too low down, the Siemens arrangement, as shown on the Glasgow retort drawing, being better in my opinion. I have recently taken out a patent for restoring the hydrocarbons to gas-coke, by dipping it in crude petroleum, which, I believe, will enable gas engineers to heat their retorts with the gas coke they have in many instances such a difficulty in disposing of. To prove what the petro-coke is capable of doing I tried some in a puddling furnace, and in an hour and three quarters produced 4cwt. of puddled bars, with 5cwt. of gas coke, mixed with 13 quarts of petroleum. As may be imagined there was an entire absence of smoke from the chimney.

MR. WEST: As gas engineers, you may be sure that we are very much interested in the question of regenerator furnaces. I have never had the opportunity before to state in public my view on this important question. Some of you are aware that Mr. Stevenson, our president, has taken a great interest in this matter, and I think we are all very much indebted to him, although he has not had a very good opportunity of getting experiments worked out himself. I may tell you that he was the first to assist me, between five and six years ago, in putting up some furnaces on the Liegel system at Maidstone. These furnaces were arranged with slits, and with the kind of coals we were then using, we managed to get very excellent heats, but we found that if we changed the coal we got into difficulties on account of not being able to run off the slag systematically, and I came to the conclusion that these furnaces would be objectionable in the Midland districts, where a variety of coal was used. At Manchester we decided to erect regenerator furnaces at our new works, and there we have to deal with various canals and coals containing from 5 to 30 per cent. of ash, and it became a question which was the best furnace to adopt. I may say that we decided to put up a number of Siemens's, some of Klönne, and some devised by myself. We have not been able yet to test the relative merits of these systems, but I may say I was led to adopt the Klönne furnaces from what I saw our friends were doing in Birmingham and other places, and I think it right to say I have been especially pleased to find that the two engineers in this town have practically solved the question. As I told you, I began very early to introduce these furnaces into England, with the assistance of our president, but was unable to bring them out so perfectly, for want of practice, as your engineers have done here. It is very gratifying to hear Mr. Hack say that he has brought down the percentage of fuel to 10lb. per every 100lb. of coal carbonised, which is proof that he has been most energetically at work and progressing in the right direction. With reference to the remarks made by Mr. Head about stopped pipes, they arise simply on account of the intense heat generated. This, however, is another subject altogether apart from regenerator furnaces, and should not be attached to it. There are one or two advantages connected with regenerator furnaces that I think have not been sufficiently dwelt upon. In some districts a saving of fuel may be of value, but in Manchester this is not worth any consideration, for at the present time we are selling coke at 3s. 4d. per ton. I attach great importance, however, to the extra volume of gas that can be produced per retort, the ease with which the firing is accomplished, and the greater durability of the retorts.

REPLY.

MR. HUNT, in replying, said he quite agreed with

Mr. West that gas-makers were greatly indebted to Mr. Stevenson for his efforts in forcing upon their attention this important subject of gas firing, and it must be very gratifying to them to know how rapidly the system is now spreading, and with what good results. He likewise endorsed what Mr. West had said in reference to the cause of stopped pipes. That this was very much a matter of high heats he had no doubt whatever, any more than he had as to what were the principal remedies to be applied, namely, moderate heats and speedy removal of the tar from the hydraulic main. No system of heating could claim immunity from stopped pipes. He had been very interested in hearing of the new use for gas coke developed by Mr. Casson, but presumed that his process was not applicable to the manufacture of illuminating gas. As to the difference in price between Manchester and Birmingham gas coke, he was of opinion that the Birmingham consumer had the best bargain, although paying 10s. per ton as against 3s. 4d., because the Manchester coke was largely made from cannel, and was consequently of a quality that he was afraid would in this district scarcely command a sale at any price. With reference to the exception taken by Mr. Head to his compound of the Didier with the Siemens generator, all he had to say was that there was an exact similarity between the two, in so far as both were hearth generators. Beyond that he had not expressed any opinion. He was glad to hear that Mr. Head was turning his attention to placing the producer within the setting. Hitherto the Siemens system had been identified with the detached generator, as shown upon the drawing. Mr. Head's memory was accurate when he said that one of the difficulties experienced in the working of the original Siemens system at Windsor Street was the accumulation of water in the flues; but this had nothing to do with the ultimate decision in regard to the system, it having been overcome during the latter part of the trial. The explanation sought for by Mr. Paterson, as to the different proportions of combustible gases existing in the Saltley and Glasgow furnaces, was to be found in the fact that with the latter steam was largely employed. He was of opinion that the proportion of CO<sub>2</sub> existing in the furnace gases was very moderate, for if he recollected rightly about 8 per cent. was the quantity shown to be present in the gases from the Schilling generator. He could not accept Mr. Stevenson's suggestion as accounting for the presence of so large a percentage of free oxygen in the chimney gases from the Saltley furnaces. Any leakage in the flues was not difficult to detect, and he adhered to the opinion that the excess was due to imperfect regulation of the air supply. He contended that it was absolutely impossible to settle the question of economy in regard to gas firing without recuperation in any other way than by applying the gas as he had done in the manner described in his paper.

## Glasgow and West of Scotland Section.

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Vice-chairman: Professor Ferguson.

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Professor Crum Brown.  
J. Christie.  
Dr. John Clark.  
Professor Dittmar.  
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R. R. Tatlock.  
Dr. William Wallace.

### Hon. Treasurer:

J. J. Coleman, 45, West Nile Street, Glasgow.

### Hon. Secretary:

Dr. James J. Dobbie, Chemical Laboratory, University of Glasgow.

## MEETINGS FOR 1881.

MARCH 11TH.—(1) On the Effect of Temperature in Dyeing. By Professor Mills and Mr. Rennie. (2) On the Production of Ammonia from the Nitrogen of Minerals. By Mr. Beilby. (3) On the Distribution of the Gases in the Lenden Chamber. By Mr. Mactear.

APRIL 8TH.—(1) On Algin. By Mr. Stanford. (2) On some Points in the Technology of Oil of Vitriol. By Mr. Mactear. (3) On Nickel Vessels for Chemical Operations. By Professor Dittmar.

APRIL 29TH. (Special meeting).—Discussion on International Methods of Testing and Sampling. Introduced by Mr. Tatlock.

MAY 13TH.—(1) On Different Methods of Producing Cold Artificially. By Mr. Coleman. (2) On the Composition of Tobacco. By Dr. Clark.

## Journal and Patent Literature.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*A New or Improved Composition for Preventing and Removing Incrustation in Steam and other Boilers.* S. Lattuada, Milan, Italy. Eng. Pat. 2747, June 2, 1883. Not proceeded with.

"I MIX together about 7 parts by weight of sal-ammoniac, 4 parts of gambier or catechu, 1 part of powdered gall nut, and 1 to 2 parts soda. This mixture applied in about 1,000 times its weight of water prevents formation of calcareous or saline incrustation, and has the effect of detaching such incrustation, and also of preserving the metal against corrosion."

*Improvements in the Manufacture of Acid-proof, Porous Masses of Clay, permeated with finely-divided Carbon, suitable for Filtering and Refining Operations.* Wilhelm Olschewsky, Berlin. Ger. Pat. 23254, Nov. 24, 1882. Addition to Pat. 19589, Jan. 31, 1882.

FOR the preparation of acid-resisting masses for purposes of filtration, very porous clay, granulated in accordance with the directions contained in the original patent, is warmed with concentrated hydrochloric acid, until the extraction of all soluble constituents is completed. The sufficiently hard and very porous unattached portion is washed and dried, and then impregnated with tar, liquid glue, or petroleum residues, which, on heating, in the absence of air, cause the clay mass to become permeated with finely-divided carbon.—W. D. B.

## II.—FUEL, GAS, AND LIGHT.

*On Kumbert's New Petroleum Lamp and B. Hoff's "Multiplier."* Dingl. Polyt. Journ. 250, 409.

THE committee of the Russian Chemical and Physical Society have awarded to Kumbert the premium for a

with that obtained from America. Hoff's multiplier is an apparatus for distilling lamp oils. Petroleum, when heated above its boiling point, is decomposed into volatile hydrocarbons containing but little carbon, and others less volatile and richer in that element. By increasing the temperature the latter undergo a similar change, and in course of time the whole of the original liquid may be converted into lighter oils and residual carbon. The construction of the multiplier will be understood on reference to the annexed figure. From the still *a* the outlet pipe *c*, which is equal in length to one-third of the height of the still, and enlarged at *d* to a globe with a diameter of one-third of the length of the pipe *c*, communicates with the cooling pipe *e*, 7 metres in length, and terminating in the water-cooled condenser *f*. From *f* the tube *g* carries away the condensed products to the collecting chamber *h*, from which the flue *k*, passing through the roof of the building, draws off inflammable gases. The proportions of the still head are so arranged that vapours of oils with a specific gravity higher than 0.83 are there condensed, while the lighter products pass over into the condensing tube. The decomposition of the petroleum sets in at a temperature of 300°. It is essential that the fire be so maintained that the fuel in the still is thoroughly boiling. The advantages claimed for this still are that good lamp oil of specific gravity 0.81 to 0.83 may be readily obtained to the extent of 95 per cent. of the petroleum taken; and since the oil from the waste of the sulphuric acid refinery may be entirely recovered by it, an important reduction in the cost of producing lamp oil is effected.—W. G. M.

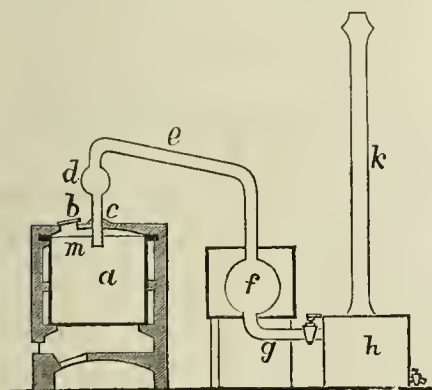
*Improvements in Coke Ovens.* Dingl. Polyt. Journ. 250, 456.

THE apparatus represented in Figs. 1, 2, and 3 has been patented (Ger. Pat. 21455, 1882) by A. Hiltawski. The slack to be coked is fed into the chambers *A* through the openings *a*. The evolved gases pass the openings *C* into the side chambers *D*, thence through *e* into *f*, lastly through *g* into the main flue *h*, whence they pass to the chimney. The draught is regulated at *e* by dampers to which access is given through the openings *O*. The openings *S* in the floor of the coking chambers *A* also serve as outlets for the gases. These are cleared from time to time during the process by means of rakes worked through *v*. The openings *e* are similarly cleared through *z*, the cinders being raked out through *a*. The supply of air for the complete combustion of the smoke is regulated by dampers *k* in the front and hind walls of each gas chamber.

C. Sachse (Ger. Pat. 22111, 1882), Figs. 4 and 5, constructs the roof of horizontal ovens with an interrupted slit. Into this, heavy slabs are introduced, which, by pressing on the charge of coal, cause the production of a dense coke. In a later patent (22826, 1882) a second slit is provided, through which the charge may be introduced without having to remove the slabs. This second slit is closed air-tight during the coking. The arrangement of the flues *m*, *n*, *o*, is explained by the drawing. The gases pass down the vertical flues *k* into *m*, thence through *u* and *o* into *v*.

In the shaft coke oven, Fig. 6, of E. Franzen (Ger. Pat. 21867, 1882) the gases evolved in the coking chamber *H* pass through the openings *e* into the vertical canal *e*, where they are mixed with atmospheric air, drawn in by way of *a* and *s*, *a* being in direct contact with the outer air. The products of combustion pass by way of *G* into the chimney.

F. Lürmann (Ger. Pat. Suppl. 17203, 1881), Figs. 7, 8, 9, and 10, has adopted in continuous coking ovens a slit, as at *S*, Fig. 10, in the floor of the coking chamber *A*, in close proximity to the door *F*. This slit is cleared from time to time by raking. Through *S* air can be admitted directly into the centre of the coking mass. Instead of passing the evolved gases through a single opening *z*, and then determining with the air in *C* an intense combustion at a point where the heat is least required by the coking mass, other openings are provided as at *a*, by which means the heat is caused to do work on the coal and prevented from acting injuriously on the material of the oven. Where the method of separating the condensable products of

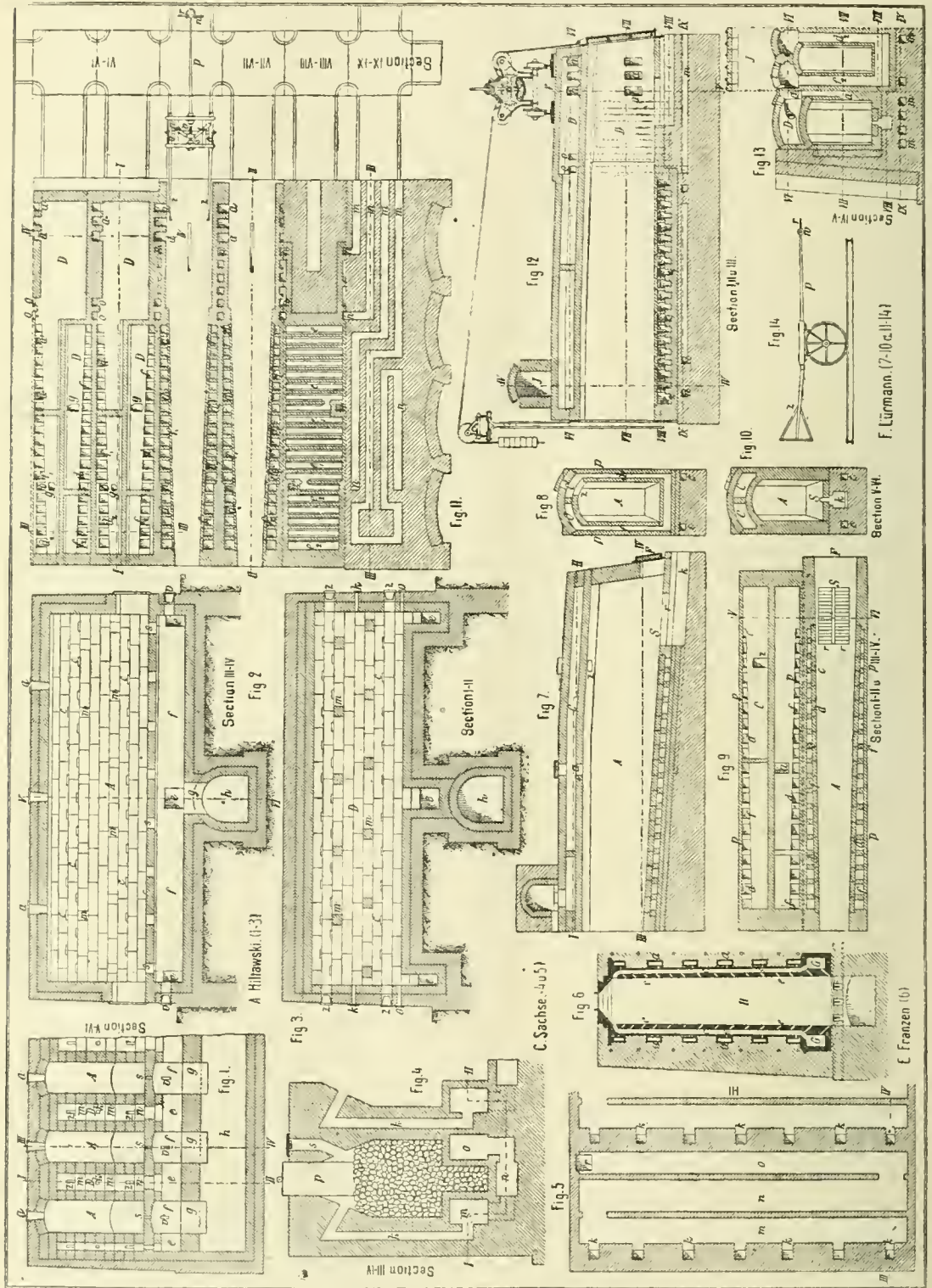


lamps suited for use with the oils of specific gravity 0.865 to 0.875, obtained by distilling Caucasian petroleum. It is hoped that this invention may enable Caucasian petroleum to compete successfully in European markets



combustion is practised, the gases, in being returned for combustion, should likewise be allowed to enter C at more than one point.

occur at the point of discharge. The arrangement which it provides consists in openings *a* in the side walls for the exit of the gases. This causes an accumulation of gases



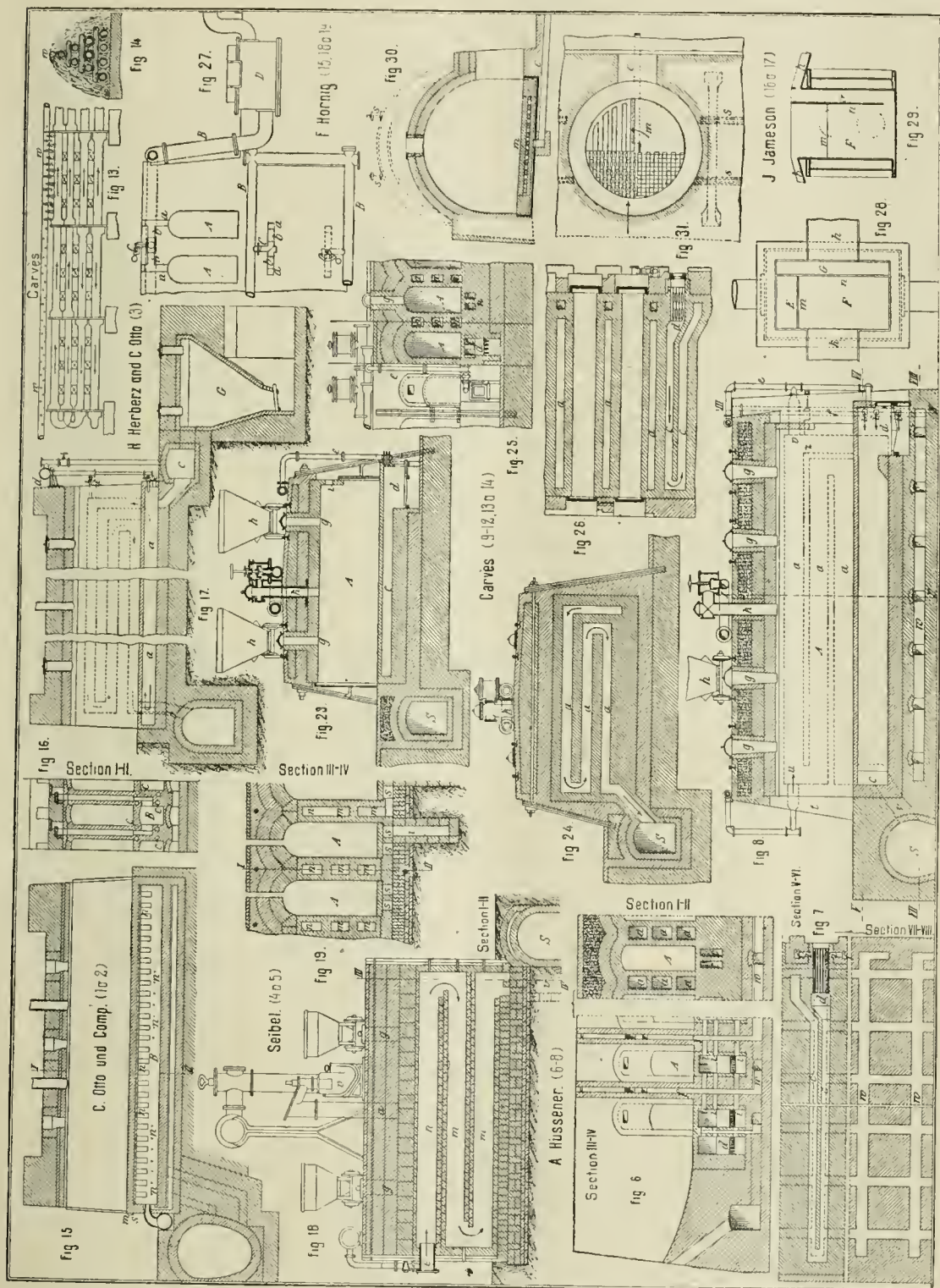
A further patent (Ger. Pat. 202111, 1882) is directed towards preventing the ingress of air into the coking chamber of continuous ovens, which is known to

in the parts which contain no coke, and being under a certain pressure they either altogether prevent the ingress of air or by combining with the air that does enter



prevent it attacking the coke. The combustion of the gases takes place in D by means of air admitted through *m* and *o*, the product of combustion passing by way of

be provided in the roof of the chamber. Lürmann's extracting tongs (Figs. 11, 12, 13, and 14), for removing the charge from coke ovens, is a locomotive apparatus.



*i* into *j*. The down draughts are indicated by *d d*, the up draughts by *f f*, the horizontal by *e e*. In addition to the gas exits *a* in the side walls similar exits *g* may

It consists of a handle *p*, with a ring at *r*; *p* terminates at its other extremity in the screw *v*, working in union with the screw *s*. The levers *t* are connected with *z*, the



extracting tongs proper. The handle *w* being turned to the left, the screw *s* is advanced along the thread *v*, and the tongs are opened by means of *t*. The apparatus is then brought to the door of the oven to be emptied, the tongs are pushed home, and by the reverse turn of the handle *w* caused to grip the mass of coke, which is then drawn by any suitable mechanical means applied at *r*.

C. Otto and Company (Ger. Pat. 20908, 1882), Figs. 15 and 16, have devised an arrangement with the object of introducing air at any required point, which consists of small passages *C* (Fig. 16) in the floor, roof, and sides of the main flue (Sohlkanal), the entrance of the air being controlled by stops *s* at the extremities *m*.

H. Herber and C. Otto have patented (Ger. Pat. 17873, 1881) an arrangement which dispenses with the auxiliary stoves hitherto used in the case of ovens adapted to the recovery of the volatile by-products. According to their plan, a sufficient number of generators *G* (Fig. 17) are worked in connection with a group of ovens, and the hot gases from the generators are conducted through *C* into *a* of the ovens; here they meet with the gases returned through *d* from the condensation apparatus and with air admitted through *e*.

L. Chenut describes a coke oven (*Ann. Ind.* 1883, p. 369), Figs. 18 and 19, worked by Seibel, in Campagnac, which is said to yield 73 per cent. coke from a coal yielding only 64 per cent. by the crucible test. The ovens *A* (Fig. 19) are 6m. long, 0·7m. wide, and 1m. high. They are charged through *g* with 5 tons of coal. The evolved gases pass through *a* to a common chamber *v*, where the condensable products are removed, the gases being returned by way of *c* to *u*. This arrangement does not appear to be essentially different from that of T. Semet and Solvay (*Dingl. Journ.* 248, 212). The yield moreover is probably over-estimated.

An exhaustive discussion of recent improvements in coke ovens will be found in *Stahl und Eisen*, 1883, p. 397, from the pen of A. Hüssener. Details are given of the working of nine small Lürmann ovens—previously described—during a period of 17 months ending March, 1883. A mixture of equal parts of cannel (Westphalian) and anthracite was employed, 4573·5 tons of this mixture yielding 3660·5 of coke, *i.e.*, 80 per cent. The dimensions of these ovens are 9m. by 0·5m. by 1m. high. Objections appear to be taken by certain operators to the quality of the coke and the labour of drawing the charge in the case of these ovens; but as these have to do with the quality of coal employed and the mechanical details of working, these defects, it is suggested, may be readily overcome, and the oven is recognised as, on the whole, a decided improvement on previous forms. The following details are given of the working of the Sachse oven, previously described, during a period of 32 days. To show the effect of weighting the charge, a parallel experiment was made in which the weighting was omitted. Results—

	Aggregate Coke.	Large Coke.	Small Coke.
With weighting . . . .	65·8 composed of 80·62	19·37	
Without weighting . . .	61·5 composed of 69·06	30·91	

The advantage of weighting the charge is thus established. A series of experiments with the Carvès oven, conducted by Hüssener and K. Möller, has led to the foundation of a Coal Distillation Company, working with 50 of these ovens, built expressly, and on a somewhat improved plan, at Gelsenkirchen in Westphalia. The original form of these ovens is sufficiently explained by Figs. 23 to 26. The improved form is set forth in Figs. 20 to 22. The retorts are 9m. long, and of conical form. In the middle they are 0·575m. broad by 1·8m. high. They hold a charge of 5½ tons of slack. It was found advantageous to use a cannel coal. The following are the details of the work which has been done by these ovens, in continuous operation, since November, 1882. Output:

	Per cent.
Large coke . . . . .	75·0
Small coke . . . . .	0·8
Dust . . . . .	1·2
Tar . . . . .	2·77
Ammonium sulphate . . .	1·10

The tar obtained was limpid, and yielded on distillation 8·8 per cent. of volatile liquids, 39·5 per cent. of pitch,

with 1·7 per cent. loss. See this Journal, vol. II. [12], page 500. The further treatment of the distillate gave the following:—

	Per cent.
Benzol (b.p., 80° to 100°) . . . . .	0·59
Benzol (b.p., 100° to 110°) . . . . .	0·49
Naphthalene . . . . .	0·39
Phenol . . . . .	1·39
Anthracene . . . . .	0·95

These ovens have been adopted in this country, and a description of 25 of these erected at Crook (Durham) is given by R. Dixon in *Iron*, 1883, 22, 260. See also this Journal II. [10], page 404. The cost of erection is given at £5,500. In 215 days' working 7,042 tons Durham coal yielded 5,424 tons 11 cwt., or 77 per cent. coke, 2·8 per cent. tar, and 12·6 per cent. gas liquor.

C. Demant (*Rev. Univ. d. Mines*, 1883, 13, 593) gives the following details of the working of the Semet ovens: 145 tons of dry coal yielded 115·3 tons of large coke, 0·98 of small, and 1·58 of dust, together, 114·5 tons, *i.e.*, 79 per cent. The quantity of tar from the same was 2,719 kilos.

F. Hernig has patented (Ger. Pat. 21908, 1882) the apparatus for collecting the tar and ammonia from the distillation of coke represented in Fig. 27. The exits *a* from two ovens, placed side by side, are connected by a cross-canal *b*, containing a two-way valve *c*, by means of which either oven can be brought into connection with *B*, and through this with *D* and a series of condensers. The condensed liquids flow through *E* (Figs. 28 and 29) back again into *D*, thence by way of *m* into *F*, where, in consequence of the heat, a more complete separation of the tar is effected. The tar flows under the partition wall *n* into *G*, and thence by way of *h* to the store tank. The ammonia water flows through *k* into a second tank.

Lastly, notices are given of papers by J. J. Jameson on this subject in *Engineering*, 1883, 36, 255, and in this Journal, 1883, p. 114; also II. [10], p. 405. In plan and section the Jameson ovens are shown in Figs. 30 and 31.—C. F. C.

#### IV.—COLOURING MATTERS AND DYES.

##### *Improvements in the Manufacture of Laundry Blue.*

M. H. Hargreaves and T. L. Hargreaves, Hull, and J. E. Hargreaves, Freshwater. Eng. Pat. 2524, May 21, 1883.

LAUNDRY blue is to be manufactured by "grinding together ultramarine, gum arabic or acacia, and sanitas." For the latter ingredient, "terebene, carbolic acid, borax, salicylic acid, or other disinfectant of like nature" may be substituted.

##### *Improvements in the Manufacture of Tannic Black, and its Application for Paints and other Useful Purposes.*

W. G. Gard and T. H. Cobley, Dunstable. Eng. Pat. 2549, May 22, 1883.

"LEATHER waste, or any other material containing tannic acid," is "heated to from 180° to 200° F.," with solution either of soda or of acetate of soda, and the liquid so obtained is then treated by a salt of iron. The "tannic black" which precipitates is then separated from its mother-liquor, washed, and dried. It may then "be ground into paint in the ordinary way," or may be employed "mixed with albumen or casein" in calico printing.

##### *Method for the Preparation of Paranitrobenzylidene Chloride.* Farbwerke vormals Meister, Lucius, und Brüning, Höchst-on-the-Maine. Ger. Pat. 24152, Jan. 5, 1883.

PARANITROTOLUENE is treated with chlorine at a temperature of 130° to 160° until the calculated quantity of the gas has been taken up. The product of the reaction is first washed with water, then with caustic soda, and finally again with water, and then recrystallised from alcohol. The paranitrobenzylidene chloride thus obtained forms paranitrobenzaldehyde on treatment with concentrated sulphuric acid.—F. M.

*Method for the Preparation of Nitro-leuco Bases of Triphenylmethane, its Homologues and similar Hydrocarbons.* Farbwerke vormals Meister, Lucius, und Brünig, Höchst-on-the-Maine. Ger. Pat. 23784, Jan. 5, 1883.

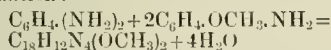
THE nitro-leuco bases are obtained from aniline, toluidine, xylidine, naphthylamine, methylxylidine, and diphenylamine by treating these bases with paranitrobenzylidene chloride, paranitrobenzylidene bromide, paranitrobenzylidene diacetate or paranitrobenzylidene glycolic ether, in the presence of a solvent such as petroleum spirit, benzene, alcohol, etc., at the boiling heat of water. Similar leuco bases are obtained from paranitrobenzylidene amide, toluidine, or xylidine by heating these compounds with the salts of the bases mentioned above in presence of a solvent.—F. M.

*Modifications in the Method for the Preparation of Artificial Indigo.* Badische Anilin und Sodafabrik, Ludwigshafen-am-Rhein. Ger. Pat. 23785, Jan. 13, 1881. Addition to Pat. 21592, Aug. 12, 1882.

THE orthonitroacetophenone described by Gevekoht (*Bericht d. deutschen chemischen Gesellschaft*, 15, 2084) is in the same manner as acetophenone converted into brominated or chlorinated substitution products which contain the halogens in the methyl group. By suitable reducing agents these substitution products can be converted into indigo blue.—F. M.

*Method for the Manufacture of Red Colouring Matters by the Action of Paradiamines upon the Amido Ethers of the Phenols.* Kalle and Company, Biebrich-on-the-Rhine. Ger. Pat. 24229, Oct. 27, 1882.

IN the same manner as safranine is formed by the oxidation of a mixture of para-diamines and primary monamines of the benzene series (aniline, toluidine, etc.), similar colouring matters can be obtained by combining para-diamines with the amido-ethers of the phenols under the influence of an oxidising agent. One molecule of the para-diamine and two molecules of the amido-phenol ether in aqueous solution are treated with potassium bichromate or another oxidising agent. The colouring matter formed is precipitated with salt. In the case of paraphenylenediamine and anisidine being used the reaction seems to take place in accordance with the following equation:—



In place of the para-diamines, their substitution products (dimethylparaphenylenediamine, etc.), or such compounds which by reducing agents are converted into para-diamines—for instance, nitrosodimethylaniline and imidodichloroquinone—may be used. In this latter case the employment of oxidising agents is unnecessary, as these bodies act themselves as such. Of the amido-phenol ethers the ortho compounds (orthoanisidine, orthoamidophenetol, orthoamidobutylphenol, etc.) are the most important; they can, however, by one half, be substituted by the corresponding para compounds. One half of the amidophenol ether may likewise be replaced by other monamines, such as aniline, toluidine, or dimethylaniline. Colouring matters of this class are prepared by the oxidation of a mixture of equal molecules of the amidophenol ether and paradiamidodiphenylamine,  $(\text{NH}_2\text{C}_6\text{H}_4)_2\text{NH}$ .—F. M.

*Method for the Manufacture of a Blue Colouring Matter.* J. F. Espenscheid, Friedrichsfeld, Baden. Ger. Pat. 23422, Feb. 5, 1882.

WHEN nitrosodimethylaniline is treated in an acid solution with sulphur chloride sulphur is separated and the liquor assumes a brownish or yellowish red colour. The solution contains now a sulphur compound, from which, by treatment with reducing agents (zinc dust, iron filings, etc.), part of the sulphur is abstracted. By oxidising the colourless solution thus obtained with ferric chloride, a blue colouring matter is formed which can be separated by adding common salt or zinc chloride to the liquor.

F. M.

*Method for the Synthetic Preparation (through abstraction of water) of Organic Compounds by the Employment of Acid Alkaline Sulphates.* Actien-Gesellschaft für Anilinfabrikation, Berlin. Ger. Pat. 23775, July 27, 1882.

INSTEAD of sulphuric acid or zinc chloride, which are now being used as water-abstracting agents, alkaline bisulphates are employed, whereby some reactions proceed much more smoothly than with sulphuric acid or zinc chloride. A number of condensations may be effected with alkaline bisulphate, in cases where sulphuric acid does not give a satisfactory result or cannot be employed at all. Potassium, sodium, or ammonium bisulphates are used in the following processes: (1) For the condensation of aldehydes with secondary and tertiary monamines, namely, benzaldehyde, nitrobenzaldehyde, oxybenzaldehyde, and benzaldehyde sulphonic acid on one hand, with methyl-, ethyl-, dimethyl-, diethyl-, diamyl-, aniline, diphenylamine, and methyldiphenylamine on the other. (2) For the condensation of aldehydes with phenols under employment of acetic aldehyde, chloral, acrolein, valeral, benzaldehyde, its nitro- and oxy- derivatives and sulphonic acid on one hand, and phenol,  $\alpha$  and  $\beta$ -naphthol, cresol, resorcin, orcin, pyrocatechin, pyrogallol, and the methyl-, ethyl-, etc., ethers of the polymeric phenols. (3) For the condensation of alcohols with phenols, such as methyl-, ethyl-, isobutyl-, amyl-, and benzyl alcohol, with phenol, nitrophenol, cresol, resorcin, orcin, pyrocatechin, pyrogallol, salicylic acid, and gallic acid.—F. M.

*Method for the Manufacture of a Black Pigment Colour from Coal-tar Pitch.* Emil Heusser, Dürkheim, Pfalz. Ger. Pat. 24231, Jan. 23, 1883.

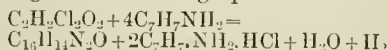
THE pitch is extracted with warm light tar-oil or benzoline. A solution is formed which on evaporation leaves a mass resembling native asphaltum. The residue left after extraction forms a black colour of great covering power.—F. M.

*Method for the Manufacture of Glass and Soluble Silicates in Pit Furnaces.* Emil Heusser, Dürkheim, Pfalz. Ger. Pat. 23950, Jan. 23, 1883.

THE glass mixture, consisting of sodium sulphate, calcium sulphate, and silica, is made up with coal pitch into briquettes, which are dried in the air. These briquettes are melted in a pit furnace under employment of a hot-air blast, whereby the sulphur is given off in the shape of sulphurous acid. The sulphurous gas is utilised for the production of sulphites of lime or soda. Soluble silicate is obtained in the same manner, the addition of calcium sulphate being omitted.—F. M.

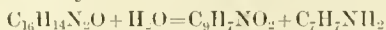
*Improvements in the Manufacture of Colouring Matters.* Paul Joachim Meyer, Berlin. Eng. Pat. 1788, April 9, 1883.

THIS invention has for its object the production of derivatives of isatin, and their conversion into indigo derivatives by the known methods of transformation. The inventor acts upon amines, such as aniline and its homologues and their alkylised or haloid derivatives, or the corresponding diamines, with a dihaloid acetic acid, i.e., by dichlor-, dibrom-, or diiod-acetic acid, in the proportion of one molecule of the acid to four molecules of the base. The reaction is effected by fusing the constituents together at a temperature of a water-bath, or by digesting the solutions in open or closed vessels. The completion of the reaction is indicated by the copious liberation of hydrochloric acid, and the product, after being washed with water, to free it from the hydrochloride of unaltered base, is crystallised from alcohol. The reaction is said to give rise to the formation (taking paratoluidine as an example of a base) of "paratoluyllised paramethylisatin—that is to say, of paramethylisatin in which the place of one atom of oxygen thereof is taken by the rest [residue?] of paratoluidine," and the inventor gives the following equation—





The "paratoluyliised paramethylisatin," on treatment with strong acid or alkali, decomposes into paratoluidine and paramethylisatin in accordance with the equation—



The inventor also gives equations showing that the ethers, amides, and aldehydes of the dihaloid acid may be used instead of the acid itself. Paramethylisatin is very similar to ordinary isatin, and can be converted into paramethylindigo, which can be used to replace ordinary indigo.—R. M.

*Modifications in the Method for the Preparation of Red Colouring Matters from Pyridine and Quinoline Bases.* Patented under 19306. Emil Jacobsen, Berlin, Ger. Pat. 23697, Dec. 16, 1882. Addition to Pat. 19306, Feb. 14, 1882.

WHEN the process described in the first patent is modified by substituting benzyldichloride, bromide, or chlorobromide for benzotrichloride—that is, when one molecule of either of these bodies is heated with two molecules of a pyridine or quinoline base and a small quantity of zinc chloride—red colouring matters are formed, closely resembling but not identical with those obtained by the former process. The colours are obtained as sparingly soluble zinc double salts, which by heating with chlorosulphonic acid or fuming sulphuric acid are transformed into sulphonic acids, the alkaline salts of which are readily soluble. These colouring matters are not produced from pure quinoline, but only from that obtained from coal tar, which besides quinoline contains quinaldine, the presence of both bodies being necessary in the formation of the colours. The best results are obtained when equal molecules of quinoline and quinaldine are heated with benzotrichloride, in which case the bluish-red colouring matter with yellow fluorescence (see Ger. Pat. 19306) is produced. The homologues of quinoline and quinaldine behave in a similar manner.—F. M.

*An Improved Method of Producing a Yellow Dye.* Gustav Adolph Bang, Leeds. A communication from Dahl and Co., Barmen, Germany. Eng. Pat. 1964, April 18, 1883.

ONE part of alphanaphtholmethyl ether is heated to 90° C. with four parts of fuming sulphuric acid containing from 10 to 15 per cent. of anhydride till a sample, on being diluted with water and warmed with nitric acid, no longer gives a precipitate. The mixture is then diluted with ten parts of cold water and warmed with two parts of nitric acid. After nitration the mixture solidifies on cooling to a crystalline mass, which is collected, pressed, and converted into the potassium salt.—R. M.

*New or Improved Process for the Manufacture of Ulin-brown.* Herbert John Haddan, Kensington. A communication from la Banque Industrielle de Belgique, Paris. Eng. Pat. 2009, April 20, 1883.

ACCORDING to this invention the manufacture of ulin-brown is carried out by macerating the wood for several days with caustic alkalis or alkaline carbonates, at an elevated temperature, the mass being occasionally boiled. The woods specified are mahogany, birch, cedar, chestnut, oak, elm, quebracho, sumach, etc. The alkaline liquid is saturated with acetic acid, a decoction of tannic acid added, and the mixture evaporated. The inventor states that the various tannins to be added to the extracts are to be selected according to their reaction with iron salts, and he gives the details of the preparation of ulin-brown for "charging silk."—R. M.

*Improvements in the Manufacture of Colouring Matters Suitable for Dyeing and Printing.* John Erskine, Glasgow. A communication from Carl Rumpff, Elberfeld. Eng. Pat. 2411, May 12, 1883.

IN a previous patent (1225, 1881) the author described a new monosulphonic acid of betanaphthol obtained by

acting upon the naphthol with sulphuric acid at a comparatively low temperature. The new acid is betanaphthol-alphasulphonic acid mixed with a certain quantity of the betanaphtholbetasulphonic acid of Schäffer. In the former specification a method of separating the two acids was described, which depended upon the solubility of the sodium salt of the new acid and the insolubility of the sodium salt of Schäffer's acid in alcohol. In the present patent the author describes a method of separating the isomeric acids by acting upon the mixture in the usual way with diazobenzene and its homologues, diazonaphthalene, etc. Under these circumstances, Schäffer's acid is the first to combine with the diazo-salt, and the colouring thus produced is salted out and removed by filtration, the filtrate containing the required alphasulphonic acid. The amount of betasulphonic acid in the mixture is first ascertained in a sample "by well-known chemical means," so that the amount of diazo compound required to remove this beta acid can be added exactly.—R. M.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

*The Production by chemical means of Permanent Embossed Designs upon Pash and other Woolen Fabrics.* W. Spindler, Berlin. Ger. Pat. 23710, Feb. 7, 1883.

COMPOUNDS such as soda, hydrochloric acid, nitric acid, tin-salt, potassium bichromate, and calcium sulphide, which have a corrosive action upon wool, are made up with starch into paste of convenient thickness, and the fabrics are printed with such paste either by hand or machinery. The printed fabrics are then dried, steamed, and washed, whereby the embossing becomes apparent.

W. D. B.

*Improvements in the Process of Rendering Wood Celluloses, Textiles, and other Products Uninflammable and Incombustible.* F. K. de Stasicki, London. Communicated by B. Hoff, Jaroslau, Austria. Eng. Pat. 1744, 1883.

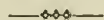
THIS invention consists in forcing "into the pores of the wood or other organic material to be treated," under a pressure of from 4 to 8 atmospheres, either a solution of 20 parts of sulphate of magnesia and 5 parts of sulphite of ammonia in 100 of water, or a solution of 15 parts of sulphate of magnesia, 5 parts of sulphite of magnesia, and 5 parts of either sulphate or chloride of ammonium in 100 of water. After having been impregnated with one or the other of these solutions, the wood or other material is to be coated with a varnish made by heating "4 parts of native red oxide of lead with 5 parts of a fatty oil," dissolving "20 parts of the soap thus obtained in 100 parts of green heavy petroleum of 0.96 sp. gr. (this is a residuum of petroleum distilling factories)," and adding to the product "20 parts of linseed oil varnish." It is said that wood so treated will "resist a heat of even 400° to 600° C., and under such conditions produces very little smoke or gas prejudicial to free breathing. Being a very bad conductor of heat, only that part of the wood which is in contact with the flame is warmed."

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Oxidation of Oil in Turkey-red Dyeing.* F. Schatz. Dingl. Polyt. Jour. 250, 9.

IN the article "On the Oiling and other Processes in Dyeing Turkey Red" (1882, 247, 38), there were incidentally mentioned steaming and hanging, but the author expressed himself in such a manner as to give rise to the construction that the less stable compounds of the fatty acids were thereby destroyed. The reason which induced him to come to this conclusion was the peculiar smell, reminding him of boiling soap-ley and slightly-burnt fats escaping from the steam-cot under certain conditions. Afterwards this conclusion was found to be incorrect. It is well known that for turkey-red dyeing the castor oil is treated

with sulphuric acid. If care be taken to wash out the glycerin completely, the above-mentioned smell is hardly at all noticed on steaming. If the glycerine be intentionally left in, or else if, as must happen sometimes in manufacturing practice, the glycerine be insufficiently removed, then the smell in question reappears, a proof that it is due to glycerine-sulphonic acid. Steaming produces an oxy-acid by separation of sulphurous acid, and a similar oxidation takes place on hanging in the air.



*Improvements in Dyeing Loose Cotton Black.* Gustav Jagenburg, Rydholm. Ger. Pat. 23357. Jan. 16, 1883. Addition to 20691, July 1, 1882.

IN place of the steaming recommended in the original patent, it is sufficient in many cases to dry, at a temperature of from 40° to 60° C., the loose cotton which has been saturated with logwood solution, finally developing the colouring matter in a cold bath under diminished pressure.—W. D. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Improvements in the Manufacture of Lime.* W. Kemp, Millers Dale. Eng. Pat. 1692, 1883. Not proceeded with.

THIS invention relates to the calcination of limestone "in what is known as the continuous drawing kiln," and consists "in charging the above-named kiln with alternate layers of fuel," and limestone "of a thickness much greater than has hitherto been the practice." The advantages claimed by the inventor are economy of fuel and "a more speedy calcination" of the limestone.

*Improvements in the Obtaining of Ammonia from Coal Gas.* W. J. Cooper, Westminster. Eng. Pat. 2669, May 29, 1883.

"IN the manufacture of coal gas there is a considerable waste of ammonia at the discharge from the condensers, owing to the strength of the ammoniacal liquor leaving the condensers." To avoid this loss of ammonia, the author of this invention proposes to feed enough water into the condensers "to dilute the ammoniacal liquor so that it is discharged from the condensers in such a diluted state that practically it will not yield ammonia to the atmosphere." The quantity of water proposed to be used is ten gallons per ton of coal carbonised.

*Improvements in the Separation and Utilisation of the Alkali used in the Extraction of Crude Carbolic Acid from Coal Tar or Coal-tar Oils.* J. Lane, Elland, and D. V. Steuart, Manchester. Eng. Pat. 2469, May 17, 1883. Void, by reason of not filing final specification.

THIS invention consists in substituting for the method of "washing coal-tar oils with a dilute solution of caustic soda, thus forming carbolate of lime, from which the carbolic acid is afterwards disengaged by sulphuric acid, soda sulphate remaining as a by-product," the decomposition of the "carbolate of soda" by sulphuretted hydrogen. The inventor proposes to treat the resulting sodium sulphide "by lime, when a sulphide of lime is formed and precipitated, and the alkali becoming disengaged and reconstituted, its solution, after settling, is fit to be again used."

*An Improved Method or Process for the Purification of Alkaline Solutions.* T. Glover, Runcorn. Eng. Pat. 2,734, June 1, 1883.

THIS invention consists in the use of native carbonate of zinc for the decomposition of the sodium sulphide contained in Leblanc soda-makers' vat liquors. The two bodies react upon each other to produce sodium carbonate and zinc sulphide.

*Improvements in the Manufacture of Hydrate of Strontia and Hydrosulphide of Sodium.* C. F. Claus, London, Eng. Pat. 2648, May 28, 1883.

STRONTIUM sulphide is to be formed by furnacing stron-

tium sulphate in admixture with carbonaceous matter. The strontium sulphide is to be boiled with water, so that a solution of it from 25° to 30° T. may be obtained. On cooling, this gives crystals of strontium hydrate, and a mother-liquor consisting of solution of strontium sulphhydrate. Three methods of treating this mother-liquor are proposed: (1) It is to be boiled down to dryness, and the residuum heated "until one equivalent of sulphur is expelled from it, and only monosulphide of strontium remains." This monosulphide is then to be treated by water for the obtainment of more strontium hydrate and more mother-liquor. (2) The strontium sulphhydrate of the mother-liquor is to be decomposed by sodium sulphate. The resulting strontium sulphate is to be treated as already described, and the solution of sodium sulphhydrate obtained at the same time is to be concentrated until it will crystallise on cooling. (3) The mother-liquor is to be treated by magnesium sulphate, the products being strontium sulphate, magnesium hydrate, and sulphuretted hydrogen.

*Improvements in the Construction of Apparatus for the Manufacture of Sulphurous Acid.* C. A. Allison, London. Communicated by the National Chemical Machine and Manufacturing Company, Newark, U.S.A. Eng. Pat. 4337, Sept. 11, 1883.

THIS invention relates to apparatus for the absorption of sulphurous acid gas by water. The apparatus described is a tall "box" formed of "two-inch yellow pine planks bolted together," and furnished interiorly with an arrangement of horizontal wooden shelves. Water enters at the top of the box, flowing over all the shelves successively, and SO<sub>2</sub> enters at the bottom of the box.

*An Improved Process for Extracting Sulphurous Acids from the Fumes of Furnaces, Ovens, and such.* E. A. Brydges, Berlin. Communicated by E. Brydges, Berlin. Communicated by E. Hänisch and Dr. M. Schröder, Rosdzin, Germany. Eng. Pat. 2621, May 26, 1883.

THE author of this invention claims that it will permit the manufacture of sulphuric acid economically from the sulphurous acid contained in furnace gases. The furnace gases are to be cooled, and then washed with cold water, in a special apparatus, of which a detailed drawing is given. "The concentration of the solution of sulphurous acid so obtained depends on the quantity of SO<sub>2</sub> contained in the furnace gases. With gases containing 1 per cent. SO<sub>2</sub>, 1 cubic metre of water absorbs 3 to 4 kilos. SO<sub>2</sub>. With gases containing 2½ per cent. SO<sub>2</sub>, 8 to 10 kilos. are absorbed." The solution of SO<sub>2</sub> is heated to 100° C., and is then converted into spray, and through this spray a current of hot air is passed. This causes the solution to give up its SO<sub>2</sub>, which passes off mixed with four times its volume of air, the mixture being richer both in SO<sub>2</sub> and in oxygen, than the gases ordinarily supplied to vitriol chambers. The waste heat of the furnace gases is used to heat the solution of SO<sub>2</sub>.

*Improvements in the Manufacture of Sulphite of Lime and of other Earthy or Alkaline Bases.* R. Powell, Liverpool. Eng. Pat. 2502, May 19, 1883. Not proceeded with.

THE method for the manufacture of sulphite of lime proposed by the author of this invention consists in "withdrawing one atom of oxygen from sulphate of lime by intimately intermingling the sulphate of lime with the necessary portion of carbon or sulphur," and heating the mixture in a closed or partially-closed vessel.

*Improvements in Deplacé's Apparatus for the Concentration of Sulphuric Acid.* Gustav Deplacé, Namur. Ger. Pat. 23159, December 31, 1882.

THE long flat boiler *a*, with rounded ends, is provided with a cover vaulted in several divisions. These vaults, or domes, are capped by bell-shaped receivers, *b*<sub>1</sub>, *b*<sub>2</sub>, *b*<sub>3</sub>,

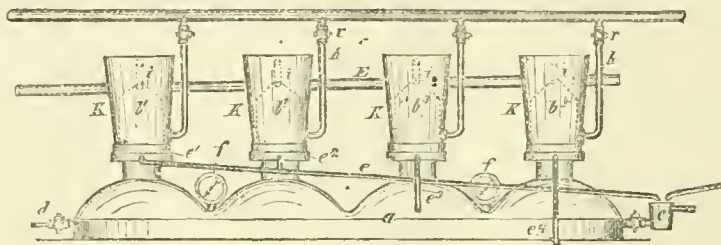
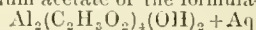


and  $b_4$ , which, from being cooled on their exterior serve as condensing heads. The acid which condenses upon and trickles down the interior of these caps serves to secure the junction between the caps and their exit openings, by which it passes into and is conveyed away by the tubes  $c_1$ ,  $c_2$ ,  $c_3$ , and  $c_4$ , blown on to the side. In the condenser head  $b_4$ , which is situated nearest to the acid supply cock C, water alone collects. This is run off by  $c_4$ . The distillate in the condenser head  $b_3$  contains, besides much water, a little distilled acid. This distillate is returned to the lead chambers. By the time that the acid passing through the apparatus has arrived at  $b_1$  and  $b_2$  its concentration has become such that a very strong acid distils into and is condensed in  $b_1$  and  $b_2$ ; from thence it flows through  $c_1$ ,  $c_2$ , and  $c_3$  into the supply funnel  $e$  and together with fresh chamber acid is again passed through the apparatus. The concentrated acid is run off at  $d$ . The temperature of the acid in the boiler

coke towers, down which are flowing the liquors, containing iron as sesquioxide. These liquors, when saturated with sulphuretted hydrogen, are mixed with a fresh quantity until potassium sulphocyanide, producing no red coloration, shows that the reduction is complete. Separated sulphur is removed from the liquor by filtration. The product obtained by the evaporation of the filtrate is pure white in appearance, and does not become of a yellow colour even after prolonged contact with the atmosphere. This is owing to the formation of the double sulphate of ferrous oxide and magnesia.—W. D. B.

*Process for Obtaining Preparations of Aluminium Acetate.* Jul. Athenstaedt. Bad Essen, near Osnabrück. Ger. Pat. 23441, Jan. 10, 1883.

BASIC aluminium acetate of the formula—



is controlled by the two pyrometers  $f$ . The boiler is placed directly over the furnace, and it is advisable to make the bottom of corrugated platinum. The employment of corrugated platinum sheet has the double advantage of strength combined with thinness, and of ready expansion and contraction proportionate to the variations of temperature.  $B$  is the inflow tube for the cooling water. The latter flows from  $B$  through the tubes  $h$ , which can be connected or shut off by the tap  $r$ , enters the cooling vessels  $K$  between their sides, and those of the condenser heads  $b_1$ ,  $b_2$ ,  $b_3$ , and  $b_4$  and finally runs away through  $i$  and  $F$ .—W. D. B.

*Process and Apparatus for the Preparation of Sulphites of the Alkalies for the Treatment of Wood and Fibrous Materials.* F. C. Kudelski, Tiffanges, Vendée. Ger. Pat. 23586, August 11, 1882.

THROUGH solutions of sodium, potassium, magnesium, or ammonium sulphate, to which calcium hydrate or carbonate has previously been added, sulphurous acid is conducted. Sulphites or bisulphites are thereby formed and calcium sulphate is precipitated. The apparatus consists of a series of vats provided with agitators, the sulphurous acid and the liquors being caused to stream through the vats in opposite directions.—W. D. B.

*Process for the Manufacture of Chloride of Lime.* Carl Opl, Hruschau. Ger. Pat. 23587, Oct. 14, 1882.

By pressure or suction, chlorine gas, previously freed from aqueous vapour, carbonic acid, and hydrochloric acid, by traversing thin layers of unslaked lime, is caused to permeate porous calcium hydrate built up in layers of convenient thickness in chambers with false bottoms. The absorption is complete when chlorine makes its appearance under the false bottoms. During the absorption the chambers are kept cool from without by means of water.—W. D. B.

*Process for Rendering the Presence of Iron Non-Detrimental to Aluminium Sulphate Neutralised with Magnesia.* F. Lienau, Uerdigen. Ger. Pat. 23493, Aug. 24, 1882. Addition to 21346, June 23, 1882.

THE sesquioxide of iron is reduced to monoxide by means of sulphuretted hydrogen passed upwards through

may be separated, from concentrated solutions, in the amorphous, soluble modification, by dissolving in such solutions, heated to a temperature of from  $30^\circ$  to  $35^\circ$  C. a sufficient quantity of sodium sulphate under prolonged agitation. The precipitation under such circumstances of the above basic acetate depends upon its insolubility in a concentrated solution of sodium sulphate. This compound can also be prepared by the double decomposition of aluminium sulphate and sodium acetate. For this end the quantity of water is so regulated that a sufficiently concentrated solution of sodium sulphate is produced to cause the separation of the amorphous basic acetate as a layer of the consistency of honey, at the surface of the liquid, which had been rapidly heated to from  $30^\circ$  to  $35^\circ$  C. The amorphous preparation soon becomes crystalline, and insoluble in water. In this form it occurs in commerce. When freshly prepared it dissolves in water, and the solution, to which a little acetic acid has been added, is well suited for dyeing operations. By mixing the crude amorphous product with lime a dry mass is formed, from which a solution of aluminium acetate is readily obtained by the addition of dilute sulphuric acid, the lime being separated as sulphate.—W. D. B.

*Process for the Manufacture of Sulphate of Soda free from Iron.* B. Schmaltz and Carl Loewig, Schönebeck. Ger. Pat. 23378, January 9, 1883.

To a hot mixed solution of kieserite and sodium chloride, of about  $20^\circ$  B., is added the solution obtained by causing concentrated hydrochloric acid to flow over sulphate of soda. The whole is filtered by means of rock salt. From the filtrate sodium sulphate, uncontaminated by iron, crystallises out. By the evaporation of the mother-liquor there is obtained a less hydrated Glauber's salt, contaminated with magnesium chloride. This crop of sodium sulphate is drained and employed in the saturation of the kieserite solution for subsequent operations.—W. D. B.

*Process for the Preparation of Barium and Strontium Compounds.* F. Muck, Bochum. Ger. Pat. 23372, Nov. 10, 1882.

FOR the preparation of barium and strontium compounds from mixtures of the corresponding chlorides with other chlorides—particularly those of sodium, calcium, and magnesium—the concentrated mother-liquor containing such a mixture is treated with a hot satu-

rated solution of sodium chloride. This dissolves all the chlorides with the exception of the sodium chloride derived from the mother-liquor. The sodium chloride liquor is repeatedly employed, and when sufficiently enriched is set aside to cool, whereby the chlorides of barium and strontium are caused to crystallise out. The mutual separation of these two compounds is effected by mixing their cold saturated solution with not much more than twice its volume of hydrochloric acid. Thereby the barium chloride is almost completely precipitated.—W. D. B.

*Process for the Purification of Solutions of Metallic Salts Contaminated with Iron.* F. C. Glaser, Berlin. Ger. Pat. 3375, Nov. 21, 1882.

The solution of aluminium sulphate, which must contain the iron as sesquioxide, is rendered feebly alkaline, and mixed with an excess of stannic acid. The precipitate produced soon subsides, and contains the iron. The supernatant solution is drawn off, the precipitate is washed, and then extracted with dilute sulphuric acid. The iron dissolves for the most part, and the residual stannic acid may be employed for repeated operations. Antimonious and antimonie acids behave like stannic acid.—W. D. B.

*Method for the Manufacture of Alkaline Hydrates and Carbonates from the Corresponding Chlorides.* La Société Anonyme Lorraine Industrielle, Nancy. Ger. Pat. 23791, Aug. 25, 1882.

THE product obtained by the action of lead oxide upon the alkaline chlorides is treated with alcohol; the solution is filtered, and the alcohol recovered from it by distillation. The residue from the alcoholic solution is freed from alcohol by the addition of a small quantity of water, and then evaporated to dryness; or the alcoholic extract is treated with carbonic acid, whereby the potash is separated as carbonate, which is filtered off. The alcoholic filtrate is again used in the process without undergoing any purification. The preparation of sodium carbonate is a little more complicated, inasmuch as this salt is slightly soluble in alcohol. The alcohol coming from the filters is therefore treated with lime, whereby the carbonate is converted into hydrate which remains in solution. The lead chloride obtained in the process is dissolved in hot water, and the lead precipitated with white pig-iron, to be again converted into oxide.—F. M.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*Process for the Ornamentation of Porcelain Articles with Dull Gilding to Resemble Chasing.* Gebrüder Schoenau, Huttensteinach. Ger. Pat. 23451, October 19, 1882.

THE plaster-of-paris moulds for these articles are cast upon models with ornamentation, possessing a finely-granular surface, either intuse or elevated. The ornamentation of the porcelain articles will therefore likewise possess a granular surface. After the biscuit-baking, the granular portions are brushed with oil, and the objects glazed by dipping, whereby the oiled surfaces are left untouched. These are now dusted with dry pulverised enamel, which, by gentle rubbing, is easily caused to enter the hollows of the ornamentation. After being baked, the objects are gilded, and now, by the difference between the gilding of the plain and that of the roughened or granular surfaces, the appearance is suggestive of chased gold.—W. D. B.

*Process for the Production on Glass of Tinted Transparent Designs on Deadened Surfaces.* S. Reich and Company, Vienna and Berlin. Ger. Pat. 23351, Nov. 5, 1882. Addition to 21540, July 18, 1882.

THE whole surface of the object is first deadened, and the design painted thereon with the compound specified

in the original patent, but, moreover, containing the necessary colouring matter. By the fluxing of this mixture transparent coloured designs are produced on a dead ground.—W. D. B.

*Method for the Manufacture of Porous Earthenware from a Mixture of Clay and Naphthalene.* Siegfried Stein, Bonn. Ger. Pat. 23947, Dec. 1, 1882.

THE naphthalene is mixed with water or dissolved in a suitable manner, and in this form well incorporated with the clay. The articles formed from this mixture are dried and then, in order to recover the naphthalene, heated to a temperature at which the latter is melted out or volatilised, whereupon they are burned in the usual manner. The burned articles show an even porosity, and as the naphthalene does not leave any ash the pores cannot inside be covered with a more fusible mass, which would partly fill up the pores, and thus make the articles less capable of resisting changes of temperature.—F. M.

*Method for the Manufacture of White Lime Alumina Glass.* Otto Korschelt, Dresden. Ger. Pat. 24227, Oct. 4, 1882.

THE glass mixture contains no alkalis, and is composed of from 8 to 10 parts of alumina, 55 to 67 parts of silica, and 35 to 15 (?) parts of lime (magnesia or baryta). The alumina is used in the shape of clay, as free from iron as possible, or as felspar-like rock rich in alumina. The lime employed may either carbonate or hydrate. The addition of silica is only necessary in case the clay employed does not already contain a sufficient quantity of it. The mixture is melted in a continuous Siemens furnace. A clear, homogeneous, strongly-refractive glass is obtained, when from 5 to 7 per cent. of the above mixture are replaced by the same quantity of soda.—F. M.

*Method for the Manufacture of Porcelain Presenting a Marble-like Appearance.* Morgenroth and Company, Gotha. Ger. Pat. 24153, Jan. 12, 1883.

THE white and coloured porcelain masses are dried till they can easily be granulated, whereupon they are passed through a wide mesh sieve. They are then moistened with a mixture of petroleum and rape oil, and mixed. The mixture is pressed in steel moulds.—F. M.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Improvements in the Manufacture of Artificial Stone.* F. H. F. Engel, Hamburg. Communicated by E. Murjahn, Hamburg. Eng. Pat. 2535, May 21, 1883.

"THIS invention has for its object the manufacture of artificial stone out of clay and peat, which stone can be made of different degrees of hardness. The process of manufacture consists of mixing in a damp state clay and peat (which latter must be pulverised in certain proportional quantities), and by forming out of such mixture stones, blocks, ornaments, or other objects, which after being dried to a certain degree are burned in kilns, or otherwise, like common bricks or pottery. According to the proportion of peat included in the mixture, the material produced varies in density, weight, and hardness. One portion of peat and two portions of clay produce a material that may be usefully employed in the construction of walls for buildings. As the weight of this improved artificial stone is less than that of common bricks, the foundation need not be so strong as would be required if the latter were used."

*An Improved Metallic Cement.* J. McConnell, Ballymena. Eng. Pat. 2845, June 7, 1883. Not proceeded with.

FINELY-DIVIDED "black antimony" and finely-divided plumbago are intimately mixed together, and the mixture



is then added to and well stirred with molten sulphur. The resulting cement is said to be "particularly adapted for closing holes and joints in cast or malleable iron, as also for cementing metal into stonework and uniting the joints of gas and water pipes, as well as of steam piping where the temperature is not higher than the melting point of the cement itself."

*Method for Utilising Silicious Earths and Rocks in the Manufacture of Cements, for the Purpose of Imparting to them Hydraulic Properties.* Johann Bielenberg, Chemnitz. Ger. Pat. 24038, Nov. 28, 1882.

SILICIOUS earths and rocks such as argillaceous porphyrite, slate, sandstone, and clay, or even mud from rivers and the sea, are ignited and then exposed to the action of sulphurous acid. These materials after such treatment attain, in combination with lime, hydraulic properties, and thus resemble the Trass, Puzzolane and Santorine earths, which are used in the manufacture of hydraulic cements. The silicious materials which are to be ignited are mixed with coal and acted upon during the process of ignition by sulphurous acid produced from sulphur, which is, together with coal, contained in pipes placed in the draught-holes of the furnace.—F. M.

*Method for the Manufacture of White Cement suitable for making Casts.* C. Haintzel, Lüneburg. Ger. Pat. 23205, Sept. 17, 1882.

For the preparation of a cement, which with water does not become hard, but which with a silicate solution solidifies to a hard mass, pure chalk is mixed with ground quartz, the mixture being formed into tiles which are burned at a white heat. The ground product is made up with a silicate into a thin paste and cast into moulds.—F. M.

## X.—METALLURGY, MINING, Etc.

*On Metallic Alloys.* Dingl. Polyt. Journ. 250, 30.

J. DÉPIERRE and P. SPIRAL, in the *Berichte der Oesterreichischen Chemischen Gesellschaft*, 1883, p. 45, publish a paper on the "Composition of Rollers used in Printworks." According to Hauvel (*Technologiste*, 1878, p. 369) a bronze of medium hardness and of the following

authors could only obtain the following analyses of doctors:—

	Copper.	Zinc.	Tin.
Yellow French doctors analysed by Salvétat .....	78.75	12.50	8.75
Yellow English doctors analysed by Berthier .....	80.50	10.50	8.00
Yellow German doctors analysed by Elsner .....	85.80	9.80	4.90

According to the researches of the authors three classes are to be distinguished: (1) Copper with 95 to 100 per cent. of copper; (2) brass with, say, 60 per cent. of copper and 40 per cent. of zinc; (3) alloys. In Table I. the physical properties of the samples examined are stated, and the remark is made that in printing rollers, where the hardness of the metal plays a pretty important role, the chemical composition alone does not fully express all the peculiarities of the metal. These also depend on the method of hardening employed, also the manner in which the roller has been tempered. The chemical composition of the samples is seen in

TABLE II.

	No. of Samples.	Copper.	Tin.	Lead.	Zinc.	Remarks.
Copper.	3	99.11	.05	.12	.57	A little aluminium
	4	99.16	.02	.12	.58	Do. and sulphur
	8	99.13	.03	.19	.45	Do. and sulphur
	9	99.03	.03	.12	.60	Do. and sulphur
	11	98.93	Traces.	.11	.67	Do. and sulphur
	2	99.67	Traces.	.07	—	—
	14	99.40	Traces.	.48	—	—
	18	99.81	Traces.	Traces.	—	—
	23	99.52	Traces.	—	—	—
	6	60.33	.03	.68	38.68	—
Brass.	10	61.70	.08	.64	37.51	—
	20	61.41	.21	2.86	31.88	—
	22	68.60	—	.39	30.53	—
	21	58.25	—	.43	41.02	—
	19	57.68	Traces.	.12	41.11	—
	11	74.51	2.80	2.18	19.85	Contain traces of arsenic and iron
Alloys.	12	76.96	2.55	1.88	17.83	—
	7	77.63	2.58	1.91	17.16	—
	5	74.12	2.37	2.22	20.59	—
	15	79.42	1.17	1.23	14.49	—
	16	72.15	3.27	1.71	22.16	—
	24	70.10	—	0.60	28.00	—
	25	15.00	—	—	81.00	—
	13	—	—	—	—	—
	14	—	—	—	—	—
	17	—	—	—	—	—

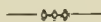
TABLE I.

No.	Colour.	Class.	Sp. Gr.	GRAIN.	HARDNESS.	REMARKS.
1	Red	1	8.82	Coarse	Hard	—
2	Red	1	8.83	Fine	Hard	—
3	Red	1	8.82	Coarse	Very soft	—
4	Red	1	8.83	Very fine	Medium	—
5	Yellow	3	8.10	Coarse	Hard	Blistered
6	Yellow	2	8.25	Very fine, regular	Hard	—
7	Yellow	3	8.58	Fine, irregular	Very brittle	—
8	Red	1	8.88	Very fine	Hard	Burnt
9	Red	1	8.80	Coarse	Soft	Suitable for printing
10	Yellow	2	8.15	Very fine	Hard	Very uneven
11	Yellow	3	8.15	Coarse, regular	Hard	—
12	Yellow	3	8.50	Fine, not homogeneous	Very brittle	Much blistered, 1835
13	Red	1	—	—	—	Very good
14	Red	1	8.90	Fine	Hard	Bad
15	Yellow	3	8.35	Fine	Hard	Very good
16	Yellow	3	8.20	Fine	Hard	Blistered
17	Yellow	2	8.10	Fine, regular	Hard	Very bad
18	Red	1	8.90	Fine	Hard	Good
19	—	—	—	—	—	—
20	Yellow	2	8.20	Coarse, not regular	Soft	—
21	Yellow	2	8.45	Fine, regular	Hard	—
22	Yellow	2	8.22	Medium	Soft	—
23	Red	1	8.85	Fine	Hard	—
24	Yellow	2	—	—	—	—
25	{ Greyish } Yellow	3	—	—	—	Attacked by colours.

composition is most suitable: Copper, 86; tin, 14; zinc, 2. Rendell obtained the following numbers on analysing an English roller: Copper, 78.3; tin, 5.6; zinc, 15.8. Although the above composition yields a hard, fine-grained alloy, yet no doubt it would be very easily attacked by the colours employed in printing. The

Besides the red copper alloys those containing 25 to 30 per cent. of zinc and 70 to 75 per cent. of copper will be found very suitable for printing. Lead, even in quantities of 0.5 per cent., acts injuriously, and all samples containing lead exhibited signs of blistering. In no sample could the presence of phosphorus be proved, and

yet the authors are of the opinion that rollers made of copper, containing from 1 to 2 per cent. of phosphorus, would work exceedingly well, both as regards resistance to chemicals and with respect to hardness, fineness of grain, regularity, and wear. Also an addition of 1 per cent. of phosphorus is recommended for the varieties of brass containing from 30 to 35 per cent. of zinc.—H. S. P.



*An Improved Method or Process of Enamelling Cast Iron.* G. J. Rhodes, Wolverhampton. Eng. Pat. 2315, May 7, 1883.

"IN applying enamels directly to the surface of cast iron as heretofore practised, when the enamel, which is applied either in a liquid state or in the form of powder, is being fired to produce a glazed surface, the said surface becomes covered with bubbles or blisters caused by the expulsion, under the action of heat in the muffle, of the gases imprisoned in the iron, thereby impairing the appearance of the enamel, and rendering it useless as a protection to the iron from rust." To prevent the formation of these bubbles or blisters, the author of this invention "expels nearly the whole of the gas from the pores of the iron, and partially fills the said pores with oxide of iron," by subjecting the iron to the Bower-Barff process before proceeding to enamel it.

*A New or Improved Process for the Extracting of Silver, Lead, Copper, and Zinc from Bluestone and other Mixed Ores, and apparatus employed therein.* J. Cross and G. I. J. Wells, Widnes. Eng. Pat. 2386. May 10, 1883.

"BLUESTONE" or others similar "mixed ore" is first digested, without being previously calcined, in hot hydrochloric acid, the lead contained in the ore being thereby converted into chloride. The liquor drawn off from the stone tanks in which this operation is performed is partly but not completely neutralised by lime, is then "well boiled," and is then filtered while hot. On cooling, the filtered liquor deposits nearly all its lead as crystals of chloride. Any lead or silver left in the mother-liquor is precipitated by "yellow liquor" from alkali waste. Or, instead of the greater part of the lead being allowed to crystallise as chloride, and the remainder being precipitated as sulphide, the whole of it is precipitated in the metallic state by the action upon the solution containing it of scrap iron. The residue left in the stone tanks in which the raw ore was treated by hot hydrochloric acid is washed, dried, and then calcined to drive off its sulphur, which is said to go off from it much more readily than from ore from which the lead originally contained therein has not been removed before calcination. The calcined ore is then treated by hydrochloric acid, which dissolves its zinc, copper, and silver. The solution so obtained is neutralised by lime or limestone or chalk, filtered, and then treated by just enough yellow liquor to precipitate as sulphides its silver and copper. These are separated from their mother-liquor by means of a filter press, and the filtrate is then treated by more yellow liquor, which precipitates as sulphide the zinc at first contained in it. Or the zinc of this liquor can be precipitated as oxide by boiling the liquor with lime.

*Improvements in the Manufacture of Fluxes or Material used in the Preparation of Plates to be Coated with Tin, Terne and other Metals.* H. F. Taylor, Neath and Briton Ferry, and G. Leghorn, Tividale. Eng. Pat. 2012, 1883.

THIS invention seems to consist in neutralising, by means of "lime or chalk or magnesia or the carbonates of any of the alkaline earths," solution of chloride of zinc which is to be used for preparing iron plates which are to be coated with tin.

*An Improved Method or Process of Extracting or Separating Iron from Solutions containing the same.* G. W. von Nawrocki, Berlin. Communicated by Loewig and Company, Goldschmieden, Germany. Eng. Pat. 2506, May 19, 1883.

THESE improvements consist in "the extraction or separation, by means of electricity, of iron from solutions of metallic salts, such as sulphate of soda or sulphate of alumina." When treating solutions of sulphates, the anode is to be of lead, "but silver is preferable for metallic chlorides." "The separation of the iron will not take place unless the production of oxygen at the anode can be almost entirely prevented. This can be effected either by using an anode with a large surface or by causing as much resistance as possible to be offered to the passage of the electric current at the anode. It is most advantageous, however, to combine both these methods." There must be no free acid in the solutions treated.

*A New or Improved Process for Producing a Protecting Coating of Rust on Cast Iron, Wrought Iron, or Steel.* L. A. Groth, London. Communicated by E. Nicolaus, Lauchhammer, Germany. Eng. Pat., May 31, 1883.

A COATING of oxide is to be produced on articles of cast iron, wrought iron, or steel by first annealing them, if necessary, then "pickling" them in dilute sulphuric or hydrochloric acid, and then, after a mechanical scouring, "so laying on a dilute inorganic acid (about 1 part acid to 5 parts of water) that the surface is covered with a very thin coating of the liquid." The articles are then to be allowed to dry completely, and then, after having been well "brushed or rubbed with clean rags," are to be again covered with a thin coating of dilute acid. After having again been allowed to dry, and "the rust which is not fixed" having been again removed by brushing or rubbing, they are to be painted over with linseed oil, and then dried in an oven.

*Improvements in the Treatment of Phosphatic Slags for the Production of Phosphates of Earths and Manganiferous Oxides of Iron.* C. Pieper, Berlin. Communicated by C. Scheibler, Berlin. Eng. Pat. 2435, May 15, 1883.

THIS invention consists in a modification of one part of the process of treating phosphatic slags which is the subject of the patents No. 5835 of 1822 and No. 1602 of 1883. The process described in the specifications of those patents begins with the reduction of the slags to a fine powder, which powder is afterwards roasted in an oxidising flame in order to convert the MnO and FeO of the slags into higher oxides. The inventor has since found that the reduction of the slags to powder before calcining them may be dispensed with. By his present invention "the slags are roasted in lumps of about a fist's thickness in an oxidising flame, and the so roasted lumps are then exposed to the action of water or steam. This has the effect that the caustic lime contained in the slag combines with the water to form hydrate of lime, and the reaction makes the lumps break up into fine powder."

*Dephosphorisation of Iron at a Red Heat.* Paul Keil, Kattowitz. Ger. Pat. 23244, Jan. 31, 1883.

THE pig iron is granulated and heated with an alkaline carbonate to a red heat. The mixture, which is contained in iron vessels, is agitated during the process.—F.M.

*Process for the Separation of Iron from Solutions obtained by the Extraction of Zinciferous Pyrites Residues.* Actiengesellschaft Georg Egestorff's Salzwerke, Linden. Ger. Pat. 23712, Feb. 22, 1883.

BARITUM or calcium carbonate is added to the solution obtained by the extraction of zinciferous pyrites residues, and air blown through the liquor. The iron is speedily



precipitated, and there is produced, on the one hand, iron, free from zinc, suitable for blast furnace operations; on the other hand, zinc salts, free from iron, are obtained.—W. D. B.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*Improved Methods or Processes for Extracting Glycerine from Fatty Substances.* W. R. Lake, London. Communicated by E. O. Banjard, Auberoillier, France.

THIS invention consists in digesting fatty substances with about 30 per cent. of their volume of water and "two or three thousandths of their weight of pure metallic zinc," very finely divided, under a pressure of "about 150lb."

*Improvements in the Manufacture of Soap.* E. A. Brydges, Berlin. Communicated by F. O. Spielhagen, Berlin. Eng. Pat. 2707, May 31, 1883. Not proceeded with.

THE author of this invention manufactures a "petroleum soap" as follows: "About 33 per cent. petroleum is mixed with about 1 per cent. spirit; about 66 per cent. fat or oil" is heated to 45° C.; the fat or oil so treated is then added to the "mixture of petroleum and spirit," and the whole well stirred. To the mixture so obtained is then added "about 50 per cent. of 40 per cent. caustic lye of soda, 25 per cent. of water glass, and 25 per cent. of silicate of alumina, infusorial earth or similar substance, and the whole mixture briskly stirred until it has grown so thick and solid that it can only be worked with great difficulty." It is then further manipulated "with forked sticks" until "it begins to steam, and is again converted into a fluid condition." It is then poured into moulds, in which it solidifies on cooling.

*Improvements in the Extraction of Gelatin, Fat, and Similar Substances from Bones, Hide, Fish, and other Animal Matter.* C. D. Ekman, London. Eng. Pat. 2680, May 30, 1883.

"THE object of this invention is to extract from bones, hides, fish, or other substances of animal origin, their useful constituents (such as gelatin and fat) in such manner as to obtain products of quality and colour superior to those obtained by the ordinary methods, and the invention consists in boiling the raw materials under pressure in solutions containing sulphurous acid with or without a base or alkali." It is preferred to use solution of bisulphite of magnesia, of such strength as to contain "¼ per cent. of sulphurous acid." The operation is performed in vessels lined with lead. When the chief object is to obtain gelatin, a pressure of 5lb. per square inch is employed; but "when the production of fat is the main object in view, then the pressure employed may exceed 10lb. to the square inch."

*The Application of Oxide of Zinc or Zinc-Dust in Assisting the Saponification of Fats by Water.* C. F. E. Poullam, E. F. Michaud, and E. N. Michaud, Paris. Ger. Pat. 23213, Nov. 2, 1882.

THE fats are heated under increased pressure in an autoclave for a considerable time with water and from 0.2 to 0.66 per cent. of their weight of oxide of zinc. The oxide of zinc may be applied either in the form of zinc-white or that of zinc-dust. It is claimed that this addition hastens the saponification of the fats and prevents discoloration of the fatty acids.—W. D. B.

*Improvements in the Manufacture of Lubricating Oil.* N. C. de Kroeber, Baku, Russia. Eng. Pat. 2123, 1883.

IN the process claimed as an invention in this patent "the residue from the distillation of petroleum—that is to say, what is left after the removal of the light and

burning oil"—is first agitated with about 10 per cent. of its weight of sulphuric acid "of a strength of about 66° Reaumur." The oil and sulphuric acid are then separated, and the former is then treated by lime or by an alkali.

*Improvements in Refining and Decolouring Soap made from Cotton-seed Oil or from other Oil containing Extractive Colouring Matter, and in Valuable Products obtained therefrom.* J. Longmore, Liverpool. Eng. Pat. 2456, May 16, 1883. Not proceeded with.

SOAP is to be made from "refined or crude cotton-seed oil, or other oils containing extractive colouring matter," by "adding sufficient caustic lye to produce saponification in the usual manner," and the soap so formed is then to be separated, instead of by salt, "by the further addition of caustic alkali." The resulting lye, "which contains nearly all the colouring matter in solution," is then drawn off, and water is added until the soap "closes." The soap is now again separated by means of caustic lye, treatment alternately by water and by caustic lye being continued until the removal of colouring matter has been accomplished as far as necessary. The coloured lyes are to be treated by carbonic or other acid to separate their colouring matter, which is then to be "utilised as a dye-stuff."

## XII.—PAINTS, VARNISHES, AND RESINS.

*Composition of Matter to be used as a Paint or Dye.* Alexander Melville Clark. A communication from the Leeds Manufacturing Company, Brooklyn, New York, U.S.A. Eng. Pat. 1683, April 3, 1883. Provisional protection only.

THE composition consists of a mixture of 5 parts of picric acid, 3 parts flavine, and 12 parts nitric acid of 36° B. This mixture is boiled to dryness, and is then said to produce "an unusually fast and permanent colour," which the inventor has named "echurine." By varying the proportions of the three constituents, different shades, varying from salmon colour to greenish yellow, are formed, and are all included in the designation "echurine." The supposed nature of the action of nitric acid upon the two other components is stated in the provisional specification.—R. M.

## XIV.—AGRICULTURE, MANURES, Etc.

*Improvements in Artificial Fertilisers, and in Methods of Manufacturing the same.* J. R. Young, junior, Norfolk, Virginia, U.S.A. Eng. Pat. 2691, May 30, 1883.

NIGHT-SOIL is to be mixed with either fish-scrap, guano, or a native phosphate, and the mixture treated by strong sulphuric acid. Besides fixing the ammonia and rendering soluble the phosphates of the materials used, some of the sulphuric acid is deoxidised to SO<sub>2</sub>, and this SO<sub>2</sub> is said to deodorise the night-soil.

## XV.—SUGAR, GUMS, STARCHES, Etc.

*A New or Improved Method and Composition for Removing Incrustation from Vessels and Apparatus employed in Sugar Manufacture.* R. de Martino, Milan, Italy. Eng. Pat. 2475, June 2, 1883. Not proceeded with.

"FOR this purpose I form a compound consisting of sal-ammoniac with about 35 per cent. of powdered gall nuts and from 20 to 30 per cent. of sumach. I introduce this compound with water into the vessel that is to be cleansed, and keep the mixture boiling, by steam heat or otherwise, for about 10 hours, or longer if necessary. The incrustation thus becomes detached and divided into small fragments, which can be removed."

*Improvements in the Manufacture of Sugar, and in Apparatus therefor.* J. Görz, Berlin. Eng. Pat. 2427, May 12, 1883.

"ACCORDING to the said invention the syrup, molasses, or other liquid is heated in a vacuum by steam at a low temperature until it forms a concentrated liquor which, after boiling for a short length of time in a vessel with or without the presence of air, is subjected to the action of the electric current. This current is allowed to pass for some time through the liquid mass, which is subsequently left to settle, when, providing the temperature does not exceed  $30^{\circ}\text{C}$ ., nearly the whole of the sugar contained in the said mass will precipitate in the form of crystals, which are afterwards collected in the usual manner. The remaining sugar is extracted from the liquor by reboiling and again passing an electric current through the mass, the process being repeated until the liquid mass is thoroughly free from sugar."

*Improvements in the Manufacture of Refined Sugar, and in Apparatus to be used therein.* J. Allen, Stepney. Eng. Pat. 2750, June 2, 1883.

"THE ordinary method of refining raw sugar is to dissolve it in hot water, separate mechanical impurities from it by filtration, decolorise by passing it through animal charcoal, and concentrate in the vacuum pan to recrystallise the sugar. This process of purifying the raw sugar is costly, as it requires a large plant with its attendant expenses, and the granulation of the sugar in the vacuum pan requires great skill and care, particularly the formation of the first grains, the ultimate result depending entirely on the proper formation of the first granulation. Attempts have been made to accelerate the formation of grain by the introduction of grain sugar into the vacuum pan, either before the boiling operation or between successive boiling operations, but with uncertain and unsatisfactory results, as no provision existed for maintaining the vacuum at the moment of such introduction, and consequently the boiling was liable to cease at a moment when it was essential to maintain it." The author of this invention describes an arrangement of apparatus by which grain sugar can be introduced into the vacuum pan without breaking the vacuum therein, and in such wise that such grain sugar "is immediately disseminated evenly throughout the liquor by the ebullition of the liquor itself, and does not fall in a mass to the bottom of the pan, or gather in clots, as it would be liable to do were the vacuum broken." He claims that "if raw sugar is thus introduced into the vacuum pan, the ordinary method of refining may be dispensed with," as regards the raw sugar so introduced, "as the liquor in the pan will rapidly remove" the impurities of this raw sugar, "and, when sufficiently concentrated, will deposit pure sugar on the grains, the impurities being subsequently removed with the syrup by means of draining or centrifugal action."

*Improvements in the Manufacture of Starch.* J. Polson and J. M. Harley, Paisley. Eng. Pat. 2703, May 31, 1883.

THIS invention has reference "not only to starch used for ordinary household or manufacturing purposes, but also to starch used for edible purposes, such as is generally known as 'corn-flour,' or 'prepared corn.'" The invention consists in drying the starch "in a stove whose atmosphere is charged with moisture or steam." This method of drying is said to render the starch "mellow, and more free from that peculiar odour and taste" said to result from the old methods of drying starch.

*Improvements in the Manufacture of Elastic Waterproof Compounds.* W. Burnham, Chicago, U.S.A. Eng. Pat. 2657, May 29, 1883.

THIS invention "is based upon the adhesiveness and insolubility in water of the sap of the mangrove tree, known in Columbia as cativo." By "combining this sap

with caoutchouc in variable proportions, permanently elastic waterproof compounds" are obtained. At ordinary temperature, "cativo" is semi-solid, but it becomes fluid at  $130^{\circ}\text{F}$ . It may be mixed with caoutchouc, "either by using appropriate solvents, such as bisulphide of carbon or naphtha, or by the employment of hot kneading rolls, by means of which the mixture is effected mechanically. In the latter case, the temperature is elevated until the mixture acquires the desired degree of plasticity." "Vulcanised" products may be obtained by kneading mixtures of cativo, caoutchouc, sulphur, or metallic sulphides and oxides. Waterproof varnishes may be made by "dissolving the cativo and caoutchouc in any desired proportion—for example, three parts of cativo to one of caoutchouc—in bisulphide of carbon, naphtha, or other solvent, to which may be added linseed oil, tar, or asphalt, or either of the ingredients ordinarily employed in the manufacture of varnishes from caoutchouc."

*Method for the Separation of Non-Saccharine Substances from Beet-root Juice.* Rudolph Bergreen, Reitzsch-bei-Bitterfeld. Ger. Pat. 23603, Dec. 12, 1882.

PREVIOUS to the treatment with lime, basic magnesium sulphite is added to the juice. The liquor having been warmed, the separation is completed by the addition of a sufficient quantity of colloidal magnesia, lime or sugar-lime. The basic magnesium sulphite is prepared by acting with sulphurous acid on suspended magnesia (obtained by precipitation) at a temperature below boiling heat, or by dissolving precipitated magnesia or magnesium carbonate in neutral or acid magnesium sulphite.—F. M.

*Improvements in the Treatment and Preparation of the substance known as "Gum Tragacanth."* A. C. Duncan, Manchester. Eng. Pat. 1933, 1883.

"To improve its condition and behaviour when used in calico printing and other manufactures," the author of this invention treats gum tragacanth by drying it "slightly," then finely grinding it, and then "roasting or calcining it in hot air, or boiling or frying it in petroleum or other oil or medium" in which it is not soluble.

*The Desiccation of Starch in vacuo.* Louis Maiche, Paris. Ger. Pat. 23355, Dec. 31, 1882.

THE blocks of starch are drained, and partially dried in a drying chest. The brownish skin which forms upon the surface having been removed, the blocks, wrapped in paper, are disposed in layers within a closed vertical cylinder heated by steam, and subjected to the action of a temperature not higher than  $25^{\circ}\text{C}$ ., exhaustion being simultaneously effected by means of an air pump. With this very rapid process of desiccation all fermentation or moulding in the interior of the blocks is avoided. W. D. B.

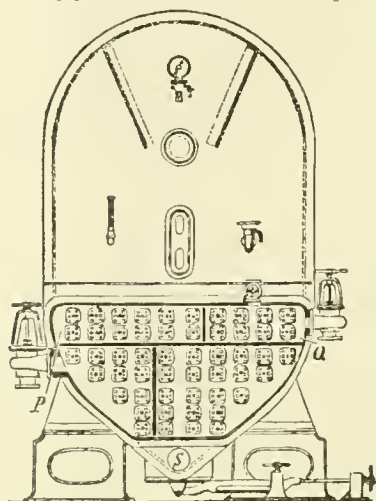
*Recovery of the Ammonia by Precipitation from the Alcohol used in the Process of Extracting Sugar from Molasses.* H. Steffens, Mühlberg-a.-E. Ger. Pat. 23594, Jan. 13, 1883.

TO the ammoniacal alcohol recovered in the distillation of the lime-sugar compound or the mother-liquors concentrated sulphuric acid is added, whereby, provided a certain degree of concentration is maintained, nearly all ammonia is separated out as crystalline sulphate. When the alcohol contains too much water, only a concentrated solution of ammonium sulphate is obtained. In this case a concentrated solution of potassium sulphate is added to the latter, taking the precaution not to bring it into contact with the alcohol. The solution of potassium-ammonium sulphate thus obtained is then mixed with the supernatant alcohol, whereby potassium-ammonium sulphate is separated in a crystalline condition.—F. M.



*Vacuum Pan with Large Heating Surface and Separate Stage Heating System for Direct and Exhaust Steam.* Franz Wellner, Pilsen, and Hugo Jelinek, Prague. Ger. Pat. 23078, Aug. 10, 1882. Addition to 6958, Feb., 1879.

THE pan has a box form, and its bottom is formed by the two slanting sides *g*. The heating chambers of the horizontal steam pipes are divided into two parts by the



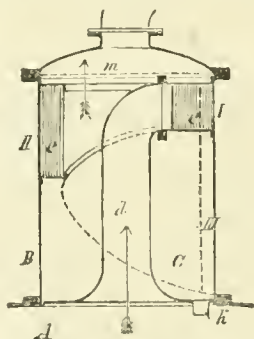
transverse wall PQ, to enable direct and exhaust steam to be used simultaneously for boiling. An endless screw S is used for removing the last portion of the contents after discharging the pan.—W. M.

*Stage Evaporating Pan.* F. Wellner and H. Jelinek, Vienna. Ger. Pat. 24001, Feb. 3, 1883. Addition to 6958, Feb., 1879.

IN order to increase the heating surface of the vacuum pan for evaporating sugar and salt solutions, while preserving its box form, the heating system is composed of several stages. The vapours from the several stages are aspirated together. The juice is either evaporated separately in each stage or run continuously from the upper to the lower stages by overflow pipes, to be run off from there for further concentration.—W. M.

*Safe for Vacuum and Evaporating Pans.* Herm. Schmidt (of the firm of C. Aug. Schmidt and Sons, Hamburg). Ger. Pat. 23606, Feb. 23, 1883.

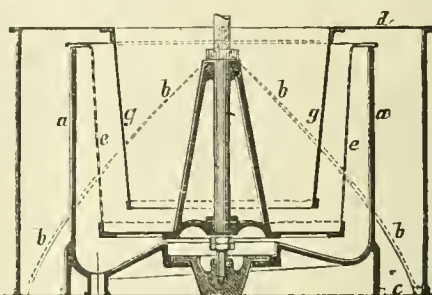
IN this safe, which is placed in the dome D of the vacuum pan A or in the overflow vessel, the steam pipe *d* is bent at the top at right angles and connected to the pipe *c*. This pipe is led in a circle round the inside of



the dome, and increases from the almost square section I. to the section II., and finally the long section III., the area remaining however unaltered. At III., after a complete turn, the pipe *c* opens in the space C, from which the steam escapes through the sieve *m*, while any liquor carried along with it falls back through *k*.—W. M.

*Modifications in Hydro-Extractors with Continuous Action.* F. W. Strohbach, Köthen. Ger. Pat. 22725, May 6, 1882.

THIS hydro-extractor, which is to serve chiefly for drying sugar crystals and extracted beetroot slices, consists of a conical basket *c* with a wide central feed-tube *g*. The outside of the casing *a* is furnished with two symmetrical channel *b b*, which surround the latter com-



pletely at the top, but diminish towards the bottom. When the hydro-extractor is working the substance to be extracted rises slowly up the inclined walls of the basket *c*, and when dry falls over the rim of the latter into the channels *b b*, from which it is delivered at the openings *c c*.—F. M.

*Improvements in the Manufacture of Glucose Syrup and Grape Sugar.* Hermann Endemann, Brooklyn, U.S.A. Ger. Pat. 24041, Dec. 24, 1882.

IN order to avoid the considerable quantity of sulphate of lime which is left dissolved in glucose syrup when sulphuric acid has been used in the manufacture, this acid is replaced by phosphoric acid, the lime salt of which is almost completely insoluble. The conversion of starch takes place under pressure, and at a temperature of 140° C., 5 per cent. of orthophosphoric acid on the weight of the dry starch being employed.—W. M.

*Process for Decolorising Crystallised Anhydrous Grape Sugar.* Th. von Korvin-Sakowicz and D. Rosenblum, Warsaw, Russia. Ger. Pat. 24181, Feb. 7, 1883.

THE starch-sugar solution, after neutralisation with chalk, is cooled to 45° C., and treated with permanganate of potassium, 0.2 per cent. on the weight of starch being used, and is filtered from the resulting brown precipitate and the sulphate of lime.—W. M.

*Purification of Beet-Juice and Molasses by means of Soluble Glass.* E. A. Schott, Kreinsen. Ger. Pat. 24129, Nov. 23, 1882.

THE raw beet-juice or molasses is treated with silicate of potash, the amount used being in proportion to the albuminous matter present, nearly neutralised with sulphurous acid, exactly neutralised with sulphuric acid, and finally filtered from the precipitated hydrate of silica.—W. M.

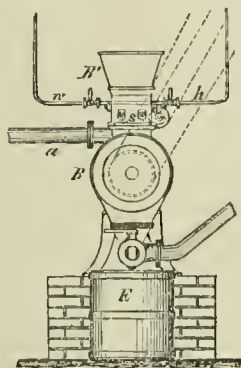
## XVI.—BREWING, WINES, SPIRITS, Etc.

*The Production of Yeast by the Fermentation of Molasses and Grains Saccharified by Means of Acids.* Gustave Claudon and Charles Vigreux, Paris. Ger. Pat. 23329, Oct. 28, 1882.

A MIXTURE of 150kg. of maize saccharified by treatment with an acid, and 2,150kg. of molasses is brought into fermentation by the addition of beer yeast, the whole being maintained at a temperature of 25° C., until the fermentation has attained its maximum. When this is the case, the temperature is reduced, by means of a cold water refrigerator, to 18° C., and kept at that point until the yeast, which began to collect during the more active stages of fermentation, has thoroughly separated. Hereupon the temperature is again raised to from 26° to 27°, and the process rapidly brought to an end.—W. D. B.

*Improvements in the Process for Preparing Mash, Yeast, and Malt-Extract.* H. Gruson, Buckau, near Magdeburg. Ger. Pat. 22621, Nov. 7, 1882.

AN "Excelsior" kibbling mill B, for grinding the substances employed in the moist condition, is interposed between the "Henze" steaming apparatus and the mash-tun F, thus providing an arrangement suitable for the production of yeast. During the disintegration of the potatoes, maize, or cereals fed into B by means of the tube A, the steam-tight cover *s* is kept closed. In the

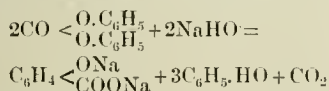


preparation of yeast material, the green malt is introduced by the funnel B, provided with a rotary toothed cylinder at its lower end, and a steady current of water at 60° C. is supplied by the pipe *w*. The pulpy yeast material collects in the vat E. For the preparation of malt-extract the same manipulation is suitable, but instead of a current of warm water at 60° C. being fed in by *w*, one of cold water is supplied by means of *k*.—W. D. B.

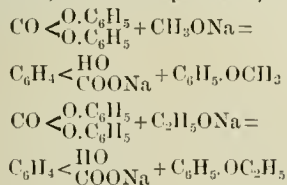
## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

*Improvements in the Manufacture of Salicylic Acid, producing also Alcoholic Phenates.* William Lloyd Wise. A communication from Dr. W. Hentschel, Dresden. Eng. Pat. 2119, April 26, 1883.

THE inventor has discovered that diphenyl carbonate can be prepared on a manufacturing scale by passing phosgene gas into an aqueous solution of sodium phenolate. This method differs from Kempf's (*Journ. of Prac. Chem.* 1, 404), in which free phenol is used and the operation performed in sealed tubes under pressure. The diphenyl carbonate is decomposed by distillation with sodium hydroxide when salicylic acid and phenol are produced—



By decomposing with sodium methylate or ethylate the phenolic ethers, anisole and phenetole, are produced—



The phenol or phenolic ether distils over, and the sodium salicylate which remains is decomposed by hydrochloric acid.—R. M.

*Paraguay Tea.* A. W. Sellin. Archiv. Pharm. 221, 292. THE author recommends this tea as being very pleasant to the taste, as well as cheaper and less injurious than Chinese teas. The *Ilex Paraguayensis*, the leaves of

which constitute this tea, is common to the mountain woods of Southern Brazil, the Argentine Republic, and Paraguay. It is an evergreen resembling the orange tree in its growth. The green twigs are pulled in April, May, and June, dried over a fire and deprived of their leaves; these are reduced by striking with wooden mallets, packed, and sent into commerce. A tree will yield 35 kilos. of the tea every three years. The total production for Brazil and Paraguay for 1880 was of the estimated value of £65,000.—C. F. C.

## XXI.—EXPLOSIVES, MATCHES, Etc.

*Blasting Cartridges composed of Saltpetre, Sulphur, Wood Charcoal, Bran, and Magnesium Sulphate.* Jean Gempeler, Vienna. Ger. Pat. 23933, Dec. 10, 1882.

SEVENTY-THREE parts by weight of saltpetre and 1 part magnesium sulphate are dissolved in one-third of their weight of boiling water, and to this solution is added 8 parts ground wood charcoal, 8 parts ground bran, and 10 parts sulphur, which components have been previously dried and mixed. The mass is thoroughly stirred and heated to 140° C. for two hours, after which it is placed in a drying apparatus at a temperature of 50° C. for five hours. The dried mass is pressed into cylinders or cartridges. The nitrate of potassium can be replaced by nitrate of sodium when the cartridges are to be used soon after being manufactured.—W. M.

## XXII.—ANALYTICAL CHEMISTRY.

*Titration of Manganese by Means of Potassium Permanganate.* Report Anal. Chem. 1883, 337, and Ber. 18, 3074.

THE solution of manganous chloride precipitated with zinc oxide is added gradually to a solution of 25 to 30 grammes of zinc sulphate, contained in a 500c.c. flask, and previously treated with a known quantity of permanganate solution more than sufficient to determine the quantity of manganese. The mixture is then made up with water to 500c.c., and an aliquot part of the solution filtered through asbestos. The determination of the excess of permanganate is effected by means of a solution of antimonious chloride according to Kessler's method. D. B.

*Qualitative and Quantitative Separation of Bismuth and Copper.* T. Löwe. Zeit. Anal. Chem. 22, 495, and Ber. 18, 3075.

THE salts of copper and bismuth are precipitated by sodium hydroxide in the presence of glycerol, the precipitates being soluble in an excess of the precipitant. From the solution the copper is completely separated as protoxide by grape sugar in the cold, the reaction being ended in the course of a few hours, whilst the alkaline solution of bismuth remains unaltered. The author bases a method for the quantitative separation of copper and bismuth on this reaction.—D. B.

*Estimation of Manganese in presence of Small Quantities of Iron.* W. Diehl. Chem. Industrie 1883, 157, and Ber. 16, 2319.

BOTH metals are precipitated by means of bromine or hydrogen peroxide and an alkaline carbonate, and the mixture of ferric oxide and manganous-manganic oxide is ignited and weighed. The oxides are then dissolved in hydrochloric acid, evaporated to dryness, and the ferric chloride decomposed in the cold with potassium iodide and hydrochloric acid. The free iodine is finally titrated with sodium thiosulphate.—S. Y.

*Estimation of Chlorine in presence of Thiocyanides.* W. Diehl. Chem. Industrie 1883, 157, and Ber. 16, 2319.

A SOLUTION containing the chlorides and thiocyanides is divided into two measured portions. In the one the



chlorine and thiocyanogen are estimated together by titration with silver. In the other the thiocyanogen is removed by precipitation with copper sulphate and sulphurous acid, the solution passed through a dry filter, and the chlorine estimated in an aliquot portion of the filtrate.—S. Y.

## New Books.

**THE ART OF SOAP-MAKING: A Practical Handbook of the Manufacture of Hard and Soft Soaps, Toilet Soaps, etc. Including many New Processes, and a Chapter on the Recovery of Glycerine from Waste Leys, etc.** By ALEXANDER WATT, Author of "Electro-Metallurgy Practically Treated," etc., etc. With numerous Illustrations. London: Crosby Lockwood and Co., 7, Stationers' Hall Court, Ludgate Hill. 1884.

AN Svo volume, bound in cloth, with an introduction of 5 pages; subject-matter covering 241 pages, and alphabetical index of 14 pages. Interspersed with the text are 36 woodcuts. Recipes are given for making various kinds of soaps, and in the closing pages of the work are tables of specific gravities and thermometric scales. Likewise there occur tables of essential oils, fusing and solidifying points of fats and oils, etc. The headings of the various chapters are as follow: Chap. I., Saponification Explained. II., The Soap Factory: its Apparatus and Appliances. III., Materials Used in Soap-Making. IV., Caustic Leys. V., VI., and VII., Manufacture of Hard Soaps. VIII., Making Soap by the Cold Process. IX., Oleic Acid: Soap from Recovered Grease. X. and XI., Cheapened Soaps. XII., Disinfecting Soap. XIII., Saponification under Pressure. XIV., Various Processes. XV. and XVI., Manufacture of Soft Soaps. XVII., XVIII., and XIX., Manufacture of Toilet or Fancy Soaps. XX., Soft Toilet Soaps. XXI., Medicated Soaps. XXII., Miscellaneous Processes. XXIII., Alkalimetry. XXIV., Analysing Soaps. XXV., Purifying of Bleaching Oils and Fats. XXVI., Recovery of Glycerine from Spent Leys. XXVII., Miscellaneous Soaps. XXVIII., Useful Notes and Tables.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1881.

1445 G. Oldfield, Leeds. Improvements in matches for lighting purposes. January 15  
1464 C. D. Abel, London. An improved manufacture of nickel and cobalt from their ores. Communicated by A. Krupp, Bernsdorf, Austria. January 15  
1472 F. Wirth, Frankfort-on-the-Main, Germany. Improvements in the manufacture of the alkaloides of quina. Communicated by Prof. Dr. Giacosa, Turin. January 15  
1485 W. R. Lake, London. Improvements in and relating to the treatment and utilisation of the residuary products of the distillation of spirits and apparatus therefor. Communicated by Dr. G. Banmert, Halle, Germany. Complete specification. January 15  
1486 W. R. Lake, London. Improvements in apparatus for use in the manufacture of sulphuric acid. Communicated by M. A. Walsh, New Jersey, U.S.A. Complete specification. January 15  
1501 F. W. Gerhard, Wolverhampton. Improvements in the manufacture of cements. January 16  
1536 C. R. Bonne, Manchester. Improvements in and apparatus for the purification of brewers' and other yeast. January 16  
1571 A. H. Huth, Kensington. Improvements in the manufacture of compounds of indiarubber, gutta-percha, and like materials. January 16  
1578 W. G. Little, Cansborough. Increasing the illuminating power of ordinary coal gas. January 17  
1639 W. R. Lake, London. An improved method or process of extracting aluminium from substances containing the same. Communicated by F. J. Seymour, Wolcotville, and W. H. Brown, New York, U.S.A. January 17  
1669 G. Valentine, F.C.S., Liverpool. Improved retorts for the production and revivification of animal charcoal from bones. January 18

1670 J. Robertson, Glasgow, and G. A. Bishop, Coatbridge. Improvements in regenerative kilns for burning bricks, pottery, or articles of a similar nature, or for calcining coal, ironstone, limestone, cement, or other substances. January 18  
1693 H. J. Bollmann Condy, Battersea. Improvements in the manufacture of white lead, and of tri-basic acetate of lead employed in the manufacture of white lead and other salts of lead. Complete specification. January 18  
1707 E. W. Harding, Bishop Wearmouth. Improvements in the manufacture of Portland cement. January 18  
1736 E. Brooke, Huddersfield. Improvements in the manufacture of a refractory material to be used for lining furnaces; also to be used in the manufacture of refractory bricks, blocks, tuyers, pipes, and similar articles. January 19  
1737 J. Trevelyan Raynes, Birkenhead. Improvements in calcining kilns and in apparatus therefor. January 19  
1738 R. Stanley and J. Sharratt, Stockingford. Improvement in the manufacture of terra-cotta and other clay goods. January 19  
1823 F. Wirth, Frankfort-on-the-Main. Improvements in the method of and apparatus for preparing and treating oils and other like substances for industrial purposes. Communicated by L. Starck, Mayence, Germany. January 21  
1834 J. S. Rigby, Widnes. An improved apparatus for the manufacture of white lead. January 21  
1843 J. C. W. Stanley, London. Improvements in the treatment of certain deposits for the purpose of recovering nitrogenous matters therefrom. January 21  
1857 F. Wirth, Frankfort-on-the-Main. Improvements in the method of preparing and using tanning substances. Communicated by L. Starck, Mayence, Germany. January 21  
1861 W. P. Thompson, Liverpool. An improved process for mixing with earthy matter and for consuming as fuel petroleum or other hydrocarbons, and apparatus for carrying the same into effect. Communicated by J. Leede, Washington, U.S.A., and G. H. Ouray, Florence, Arizona, U.S.A. Jan. 22  
1870 W. J. Grass, West Hartlepool. Tanning hides, called plutonic tanning. Communicated by W. Hassler, Hogsby and Staby, Sweden. January 22  
1881 J. H. Johnson, London. Improvements in the manufacture of Portland cement. Communicated by R. Whitman Lesley and J. M. Wilcox, Philadelphia, U.S.A. January 22  
1889 E. Askew Bath, Sketty. An improved process of treating "cotton (or like) waste" (used by engineers and others) for the reutilisation of the same, and of oleaginous or fatty matters adhering thereto. January 22  
1890 J. Harrison, Battersea. Improvements in the production and use of carbonic acid for refrigerating and aerating. January 22  
1912 P. Everitt, London. Improvements in the manufacture of gas for incandescent lighting and apparatus therefor. January 22  
1916 C. Arkoll, Chatham. Economical preparation of brewers' distillers' and vinegar makers' worts. January 22  
1932 M. Williams, Wigan. Improvements in the purification of coal gas. January 23  
1966 T. White, Thorpe Hesley, and G. Dawson, Sheffield. Improvements in the means and apparatus for the manufacture of useful products from shale or bind or the refuse of coal or ironstone mines, and the like substances. January 23  
1985 W. Fairweather, Glasgow. Improved means or process for filtering and decolorising sugar liquors, syrups, and saccharine juices. Communicated by F. Kermann, Schoeningen, Germany. January 23  
1991 A. Gutensohn, Mile End, and A. Cox, Bristol. An improved process for the recovery of indiarubber from waste carding, cuttings, and other pieces of indiarubber cloth. January 23  
1998 J. Inray, London. A new or improved process for decarbonating carbonate of strontia. Communicated by H. Leplay, Paris. January 23  
1999 J. Inray, London. An improvement in the strontia treatment of saccharine liquors and apparatus for that purpose. Communicated by H. Leplay, Paris. January 23  
2046 B. H. Remmers, Glasgow. Improvements in apparatus for separating solid impurities from liquids. January 24  
2076 H. A. Gadsden, London. Improvements in the extraction of the metal aluminium from its ores or compounds. Communicated by E. Fonte, New York, U.S.A. January 24  
2117 E. Carey, H. Gaskell, jun., and F. Hurter, Widnes. Improvements in the manufacture of hydrochloric acid and sodium carbonate from sodium chloride. January 25  
2118 E. Carey, H. Gaskell, jun., and F. Hurter, Widnes. Improvements in the treatment of sulphate of ammonia for obtaining the ammonia in the free state and utilising the sulphuric acid. January 25  
2119 A. P. Price, London. Improvements in recovering and obtaining tin from tinned metallic surfaces. January 25  
2143 G. G. Melnibridge Hardingham, London. Improvements in explosive cartridges for blasting and other purposes. Communicated by T. Woolhouse, Paris. January 25  
2146 J. G. Williams, West Hampstead. Improvements in the manufacture of hydrated oxide of iron. January 25  
2157 J. H. Johnson, London. Improvements in treating ores or scoria containing copper or nickel, and in apparatus connected therewith. Communicated by E. Hermite, Rouen. January 25  
2162 A. E. Scott, South Kensington. Improvements in treating substances and extracting metals and metalloids therefrom. January 26  
2183 R. A. Mossman, and J. Mayel Magelston, Elloughton. The application of bicarbonate of ammonia to the leavening or expansion of dough made of wheaten flour or other farinaceous substance. January 26

- 2218 G. Baron de Overbeck, London. Improvements in the production of aluminium. Communicated to him by H. Niewerth, Hanover. January 26
- 2219 G. Baron de Overbeck, London. Improvements in explosive compounds in the nature of gunpowder. January 26
- 2223 A. Pillans Laurie, Edinburgh. Improvements in extracting lead, silver, copper, and zinc, from the bluestone ore of Anglesea and similar ores. January 26
- 2229 F. Week, Lilleshall. Improvements in machinery or apparatus for washing and scrubbing coal gas. January 26
- 2217 C. J. Lortzing, Charkow, Russia. Method to manufacture asphalt compriore or asphalt mastics from the firm residuums of the outwashing waters in the wool washings, combings, fullings, and tannings, and of all industries related to them. January 28
- 2253 H. A. Morgan and Lees Taylor, Llantrissant. Improvements in the means of or apparatus for treating tin andterne plates in the process of cleaning them. January 28
- 2263 J. D. Thomas, T. Wade Evans, and J. Gravel, Swansea. Improvements in the manufacture of tin andterne plates. January 28
- 2269 Dr. O. Hoffmann, Mainkur, Germany. The manufacture of new colouring matters. January 28
- 2279 W. T. Walker, Highgate. Improvements in apparatus for purifying coal gas. January 28
- 2286 H. H. Lake, London. Improvements in and relating to evaporating apparatus. Communicated by A. Vivien, St. Quentin, and A. D. R. Dujardin, Lille, France. January 28
- 2299 T. Griffiths, Oxtou. Improvements in the manufacture of aluminium. January 29
- 2307 L. A. White, Chorlton-on-Medlock. A process for metallising wood and other substances. January 29
- 2316 A. Wilkinson, Camberwell. Improvements in the manufacture of paper, paper-pulp, paste-board, mill-board, papier-maché, and such substances applicable for cabinet and all kinds of structural purposes. January 29
- 2323 J. Hatfield, Kew. Improvements in the manufacture of artificial stone. January 29
- 2330 H. J. Haddan, London. A new or improved process for the manufacture of alcohol. Communicated by C. Coster, Bruxelles. January 29
- 2334 G. F. Redfern, Finsbury. A new method of treating the residues from soda works. Communicated by Dr. H. Von Miller, and C. Opl, Hruschau, Austria. January 29
- 2311 A. W. L. Reddie, London. Improvements in the apparatus for and process of purifying gas. Communicated by the South and Sayre Manufacturing Co., New York. January 29
- 2311 J. H. Johnson, London. Improvements in apparatus for mixing and elevating sugar in course of manufacture, or for analogous purposes. Communicated by the Compagnie de Fives-Lille, Paris. January 29
- 2351 F. W. Martino, Sheffield. Improvements in the manufacture of artificial manure. January 29
- 2360 J. F. Smith, Leicester. Improvements in the manufacture of glue and size from what has been considered useless refuse. January 30
- 2368 H. Hargreaves, Congleton, W. Beckett, Oulton, and W. Cliffe, Congleton. Constructing kilns and ovens for burning limestone and bricks and other like materials. January 30
- 2372 J. K. Kaye, Moldgreen. Improvements applicable to the dyeing of black colour upon cotton and other vegetable fibres. January 30
- 2388 J. Graham Tatters, South Shields. Improvements in the mode of obtaining or recovering hydrochloric acid. January 30
- 2412 T. E. Scheele and T. Kühn, Emmerich-on-the-Rhine. Improvements in the manufacture of artificial manure. January 30
- 2415 W. T. Walker, Highgate. Improvements in apparatus employed in the purification of coal-gas. January 30
- 2418 C. D. Abel, London. Improvements in the treatment of phosphates of iron, manganese, and aluminium with the chlorides of the alkalies and alkaline earths, for obtaining alkaline phosphates and other products therefrom. Communicated by Paul Dietrich, Berlin. January 30
- 2468 H. J. Haddan, London. An improvement in the production of malt. Communicated by H. Hackmann, Mellichstadt, Bavaria. January 31
- 2476 B. H. Remmers, Glasgow. Improvements in apparatus for cooling or heating liquids. January 31
- 2477 W. Fairweather, Glasgow. An improved filtering medium, and mode of employing the same for filtering, decolorising, and deodorising liquids and liquid solutions. Communicated by F. Kleemann, Schoeningen, Germany. January 31
- 2485 B. Midgley, Milnsbridge, and W. Nunwick, Longwood. Improvements in apparatus for use in the manufacture of gas. January 30
- 2491 T. Heskin, Preston. Improvements in the mode of producing or obtaining ammoniac chloride. January 31
- 2501 W. Wyatt, Ellesmere. Improvements in the method of carrying out the process of softening and purification of water, and in apparatus for use therein. February 1
- 2539 F. Wirth, Frankfort-on-the-Main. Improvements in paints. Communicated by O. Fischer, Karlsruhe, Germany. February 1
- 2543 E. G. Brewer, London. Improvements in distilling apparatus. Communicated by D. Haicaut, Boitsford, Belgium. February 1
- 2561 C. D. Abel, London. An improvement in apparatus for drying or desiccating substances by means of heated air. Communicated by E. Langen, Cologne, Germany. February 1
- 2568 B. J. B. Mills, London. Improvements in the treatment of excremental matters, and in apparatus employed therein. Communicated by J. M. Guenantin, Paris. February 1
- 2573 A. M. Clark, London. Improvements in the process of making malleable ferro-nickel and ferro-cobalt. Communicated by the Société Anonyme dite Fonderie de Nickel et Metaux Blancs, Paris. February 1
- 2621 E. Siddons Wilson, and A. Bannen O'Connor, Camberwell. An improved method of operating upon oily and greasy rags and fibrous material, and in recovering oil and grease therefrom. February 2
- 2630 A. Germot, and L. Riviere, Paris. An improved mode of treating certain resins for obtaining a new elastic substance. February 2
- 2638 F. Wirth, Frankfort-on-the-Main. Improvements in the method of utilising certain wasteliquids. Communicated by F. Graessler, Canstatt, Germany. February 2
- 2640 A. Guattari, Paddington. Improvements in the manufacture of marble. Communicated by M. Sodorini, Florence. February 2
- 2666 J. H. Loder, Utrecht. Improvements in the manufacture of alcohol and alcoholic solutions of colouring matters, etc. February 4
- 2693 J. B. Mackey, London. The preparation of new soluble salts of cerium for medicinal purposes. Complete specification. February 4
- 2701 C. D. Abel, London. An improved process for obtaining cellulose or woody fibre from wood or other vegetable substances, and the preparation of lye therefor. Communicated by C. F. Dahl, Dantzic. Complete specification. February 4
- 2709 B. C. Sykes, M.D., and J. Briggs, Cleckheaton. Improvements in apparatus to be employed for making producer gas, heating water, and generating steam. February 5
- 2710 J. Groome, Darwen. Improvements in composition for covering steam boilers, steam pipes, or other surfaces, to prevent radiation of heat therefrom. February 5
- 2748 S. H. Emmens, and the United Patents Corporation, Limited, London. Improvements in electrolytes and in the manufacture of chemical products therefrom. February 5
- 2765 W. Brown, Bramley. A new or improved means or apparatus employed in the treatment of leather during and after the "stuffing" operation. February 5
- 2787 A. Guattari, Paddington. Improvements in the manufacture of marble. Communicated by M. Sodorini, Florence. February 5
- 2796 W. H. Swim, Runcorn, and G. Pryde and J. Hedley, Liverpool. Improvements in the treatment of certain residual salts formed in the manufacture of caustic alkalies. February 6
- 2812 F. Iles, and C. Wakeman, Birmingham. A new composition or alloy entitled "pyrogen." February 6
- 2837 C. Holliday, and E. W. Hirst, Huddersfield. Improvements in dyeing wool or other similar animal fibre, and in apparatus employed therein. February 6
- 2843 J. Cripps, jun., Brixton. Improvements in the manufacture of size from wet calf, white leather, shavings, or cuttings. Complete specification. February 6
- 2847 A. Guattari, Paddington. Improvements in the manufacture of imitation marble. Communicated by M. Sodorini, Florence. February 6
- 2862 P. C. Buon, Norwich. Improvements in the manufacture or production of pigments. February 6
- 2877 J. Johnson, Liverpool. Improvements in the manufacture of blacklead. February 7
- 2892 A. Sadée, Bonn-on-the-Rhine, and F. Schneider, Struth, Prussia. Improvements relating to the treatment and utilisation of cellulose or wood fibre. February 7
- 2896 O. Wolff, Dresden. A process of manufacturing weather-proof oil colours or paints. Communicated by A. T. Bergh, Dresden. February 7
- 2900 Bondy Azulay, Islington. An improved process of preserving the edible parts of fish. Complete specification. February 7
- 2912 J. W. C. Holmes, Huddersfield, and B. Midgley, Milnsbridge. Improvements relating to the heating of gas-retorts, spelter-pans, or the like, and apparatus therefor. February 7
- 2941 J. Knowles Kaye, Moldgreen. A new or improved method of dyeing a fast yellow colour on cotton and other vegetable fibres. February 8
- 2942 D. G. Stansby and J. Parton, Birmingham. Dividing and thereby increasing in volume and illuminating power the ordinary gas light. February 8
- 2966 C. E. Thierry, London. Improved means of treating gelatine and the like materials or substances such as imitation ivory, or what is known as porcelain and ivorine, so as to prevent such materials or substances from curling when subjected to heat, and to render the same waterproof. February 8
- 2982 C. H. Roekner, Newcastle-upon-Tyne. Utilising the spent lime from bleaching powder. February 8
- 2996 L. Mond and G. Jarnay, Winnington. Improvements in and appertaining to the manufacture of sodium bicarbonate. February 9
- 3013 J. Dixon, Oughtibridge. Improvements in the manufacture of paper, and in the machinery connected therewith. Complete specification. February 9
- 3015 G. S. Hazlehurst, Rhyl. Improvements in and relating to the manufacture of salt from brine. February 9
- 3038 P. A. Maignon, London. An improved compound for the elimination of dissolved matter from water. February 9
- 3059 H. E. Scholefield, Manchester. Improvements in the manufacture of sulphurous acid bisulphites or sulphites. February 11
- 3066 F. W. Marsh, Middlesbrough. Making gas from mineral oils. February 11
- 3073 E. Luck, London. Improvements in distilling solutions of acetic acid or vinegar. February 11



3086 M. Finch, Silvertown, and W. Willoughby, J. Willoughby, and S. Willoughby, Plymouth. Improvements in the method of and apparatus for manufacturing sulphuric acid. February 11

3090 A. Zdziański, Brest-Litovsk, Russia. Improvements in obtaining aluminium bronze. Communicated by J. Boguski, Warsaw. February 11

3097 C. D. Abel, London. A manufacture of oxy-pyrazols, or substances formed by condensing hydrazines with the ethers of carbo-retonic acids. Communicated by the Farbwerke Vornals Meister, Lucius, and Brünning, Hoechst-am-Main, Germany. February 11

3132 F. Firth, Dewsbury. An improved means or method of cooling the brickwork of furnaces for melting glass and other materials. Complete specification. February 12

3166 F. Wirth, Frankfort-on-the-Main. Improvements in the manufacture of certain sulpho-acids, and in producing colouring matter therefrom. Communicated by O. Mühlhauser, and the firm of Farbwerk, Griesheim-a-M., Dittler and Co., Griesheim, Germany. February 12

3171 M. Ziegler, London. Improvements in the manufacture of substitutes for leather. February 12

3236 H. Haek, Saltley, and F. Ley, Barrow-on-Trent. Apparatus for receiving and conveying away coke drawn from gas retorts or coke ovens, and for delivering the same into wagons, barges, or elsewhere. February 12

3240 J. Lewthwaite, Halifax. Improvements in metals and alloys or mixtures of the same. February 13

3241 W. R. Lake, London. Improvements in the manufacture of sugar, and in apparatus therefor. Communicated by K. Trobach, Berlin. Complete specification. February 13

3249 W. R. Lake, London. Improvements in the preparation of selenium for, and the manufacture of, selenium cells for electrical purposes, and apparatus therefor. Communicated by C. E. Fritis and D. H. Hopkinson, New York. Complete specification. February 13

3255 C. J. Plather, Southport. The removal of the incrustation in steam boilers, pipes, cisterns, and other vessels. February 14

3309 W. A. Barlow, London. A new and improved system of apparatus for evaporation and distillation, and for the practical application of the same. Communicated by P. Calliburees, Paris. Complete specification. February 14

3323 F. Wirth, Frankfort-on-the-Main. Improvements in the method of and apparatus for treating acid-sulphate of soda or bisulphate of soda for industrial purposes. Communicated by H. Herberts, Barmen, Germany. February 14

3355 C. D. Abel, London. An improvement in furnaces for burning small, moist, or liquid fuel. Communicated by G. A. Godillot, Paris. Complete specification. February 15

3363 J. Elliott and F. J. Parsons, Hastings. Improvements in the manufacture of artificial stone, paving slabs, and blocks. February 15

3365 A. Thomas, West Cowes. Testing gas for impurities. February 15

3370 D. H. Dade, Oglander Road, Surrey. Improvements in non-conducting and fire-proof compositions and articles. February 15

3375 J. Salamon, Wennington. An improved process of tanning hides or skins. Communicated by L. Vergez, Tarbes, France. February 15

3378 W. Guest, New Cross. Improvements in the treatment of leather. February 15

3382 T. Carlile and J. Park, Glasgow. Improvements in the manufacture of bichromate of soda. February 16

3405 A. Wright, London. The production, distribution, and administration of spray or vapour, called the "Atmonemeter." February 16

3417 J. Nuttall, Rochdale, F. Nuttall, Oldham, and J. Rouse, Oldham. Method for purifying noxious or gaseous vapours arising from heated liquids, oily or other matters, and in apparatus used therein. Complete specification. February 16

3433 J. H. Johnson, London. Improvements in treating saccharine solutions for the purpose of extracting the sugar therefrom. Communicated by V. Four, Paris. February 16

3434 L. A. Groth, London. Improvements in the manufacture of night and other illuminating lights. Communicated by F. E. Berta, Fulda, Germany. Complete specification. February 16

3435 B. Tower, Westminster. An improved method and apparatus for condensing steam or vapour. February 16

3441 C. Hunt, Birmingham. Improvements in the arrangement of the air and waste-gas passages in connection with regenerator furnaces. February 16

3452 H. H. Lake, London. Improvements in apparatus for distilling liquids for purifying gas and for similar purposes. Communicated by P. A. Mallet and T. A. Pagniez, Paris. February 16

3465 J. Peddar, Appleton-in-Widnes. Improvements in abstracting valuable properties from solutions, and in apparatus therefor. February 18

3471 C. J. Ellis, Glasgow. Improvements in obtaining sulphate of soda, and in apparatus therefor. February 18

3483 W. Martyn, Wadebridge. Dissolving leather for use as a manure. February 18

3498 H. Barclay and E. Simpson, Harrington. Improvements in the collection and treatment of flue-dust. February 18

3500 E. W. Scofield, Brixton. Compound for sweetening or restoring sour liquids, and for other purposes. February 18

3520 J. G. Willans, West Hampstead. Improvements in carbonising coal. February 18

3523 H. Townsend, Bradford. Improvements in the process and apparatus for the manufacture of gas. February 18

# THE JOURNAL OF THE Society of Chemical Industry.

## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

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### NOTICES.

#### ANNUAL GENERAL MEETING.

The next Annual General Meeting of the Members of the Society of Chemical Industry will be held at Newcastle-on-Tyne, in July. Its exact date is not yet fixed, but will be announced in due course.

The morning of the first day of the meeting will be devoted to the transaction of the ordinary business of the Society and an address by the President, and the afternoon to a visit to a chemical work. In the evening those of the members attending the meeting who wish to do so will dine together. Members may obtain dinner tickets for their friends.

At the last Council meeting it was resolved, "That the Council desires to record its entire approval of the decision of the Newcastle Section that at the annual dinner in July there shall be two classes of dinner ticket, one for dinner without wine and one for dinner with wine."

It is proposed that the morning of the second day of the meeting shall be devoted to the reading and discussion of papers on the more important of the recently-proposed methods for utilising the condensable products obtainable from coal used in the manufacture of coke, coal used in blast furnaces, and coal used as fuel. Members who may desire to read papers on any branch of this subject are invited to communicate with the General Secretary.

Papers by Mr. W. S. SUTHERLAND and Mr. GEORGE BEILBY are already promised, and others are being arranged for. By the kind courtesy of Messrs. BAIRD AND COMPANY, those members who choose to visit Gartsherrie will be permitted to see the process at work there. Mr. BEILBY has kindly arranged that those who wish to do so may see the YOUNG and BEILBY process in operation at two works near Edinburgh; and it is expected that opportunities will also be afforded of seeing in operation both the JAMESON process and the SIMON-CARVES process. The afternoon of the second day of the meeting will probably be devoted to visits to various works in the neighbourhood of Newcastle, and the members will probably be invited to a conversazione in the evening.

Full particulars of the arrangements for the meeting will be given in the June number of the Journal.

Numerous inquiries have been made for sets of the Journal for 1882. Some of the numbers for that year are out of print. Members who have not the volume for 1882, and wish to obtain it, are invited to signify that wish to the Secretary as early as possible. If a sufficient number of applications for that volume are received, the missing numbers of it will be reprinted.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.



In this number of the Journal is inserted, as the first of a series, a list of changes which have taken place during the month in the residence and in their relation to the Society of some of the members, for the information of the Society's officers and of whomsoever else it may concern. The General Secretary trusts that members will second his efforts to make this list a complete and accurate record by notifying to him their changes of address as promptly as possible.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 36, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

#### LIST OF MEMBERS ELECTED MARCH 21st, 1884.

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 Jesse P. Battershall, Ph.D., 402, Washington Street, New York, U.S.A., chemist.  
 William Beardmore, Parkhead Forge, Glasgow, steel manufacturer.  
 R. W. Peregrine Birch, M.I.C.E., 2, Westminster Chambers, Victoria Street, London, S.W.  
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 Azariah Griffiths, Clyde Cottage, Falkirk, N.B., fireclay manufacturer.  
 Alf. Hunzinger, care of E. Potter and Company, Dinting Vale, Glossop, near Manchester.  
 F. J. Lloyd, 4, Lombard Court, Gracechurch Street, E.C., analyst.  
 Otto Lathy, 225, Church Street, Philadelphia, U.S.A.  
 Donald McDonald, Invernevis, Fort William, N.B., distiller.  
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 A. Russell Miller, The Cairns, Cambuslang, near Glasgow, chemist.  
 T. Paterson Miller, The Cairns, Cambuslang, near Glasgow, Turkey-red dyer and calico printer.  
 John Murphy, 11, Wellesley Terrace, Prince's Park, Liverpool, chemical manufacturer.  
 K. Oehler, Offenbach-am-Main, Germany, anilin dye manufacturer.  
 Andrew Pears, Lanadron Soapworks, Isleworth, Middlesex, wholesale perfumer.  
 F. W. Richardson, 40, Church Street, Manningham, Bradford, analyst.  
 J. W. Roxburgh, Levenbank Works, Jamestown, Dumbartonshire, manager.  
 Ernest Smith, 114, West Street, Glasgow, manufacturing chemist.  
 Adolph Sommer, 529, Market Street, San Francisco, U.S.A., pharmacist.  
 Charles Stephens, F.L.S., Avenue House, Finchley, ink manufacturer.  
 David Stewart, Woodside, Wemyss Bay, Renfrewshire, N.B., technical chemist.  
 Richard Tangye, Gilbertstowe Hall, Bickershill, near Birmingham.  
 Gottfried Delitsch, 30, Raeburn Place, Edinburgh, chemist and manager.  
 Robert Hamilton, jun., Westthorn, Tolleross, Glasgow, analyst.  
 Howard P. Ryland, The Cedars, Gravelly Hill, Birmingham.  
 Howard Charles Ward, Sussex Villa, Putney Park Avenue, London, S.W., barrister-at-law, deputy-chairman Gas Light and Coke Company Limited.  
 George B. Anderson, Palm Cottage, Runcorn, Cheshire, manager alkali works.  
 James Angus, Lugar, Ayrshire, N.B.  
 John Currie, 38, Bridge Street, Alexandria, Dumbartonshire, chemist.  
 John Dyson, 231, York Street, Cheetham, Manchester.  
 Thomas L. Eastlake, 23, Great George Street, Westminster, London, S.W.  
 G. H. Irvine, 2, Salisbury Quadrant, Strathbungo, Glasgow, chemist.  
 William Kennedy, 28, Royal Exchange Square, Glasgow, managing director of Broxburn Oil Company Limited.  
 James Mavor, 131, St. Vincent Street, Glasgow, drysalter.  
 James Muter, 2, West View Gardens, Dennistoun, Glasgow, assistant professor of "technical chemistry" ("Young" chair), Anderson's College.

J. Barton Scammell, 13, Lordship Park, Stoke Newington, London, N.  
 G. R. Tweedie, 13, Chantry Road, Brixton, London, S.W., analyst.  
 J. H. Wainwright, 402, Washington Street, New York, U.S.A.  
 Hugh W. Walker, Greenock, N.B., sugar refiner.  
 Theodore Wehler, 32, Liberty Street, New York, U.S.A., manufacturer of percussion caps.

#### CHANGES OF ADDRESS.

Barnes, R. L., Lancaster House, Savoy, W.C. (late of Buckhurst Hill).  
 Clark, E. B. (late of Glasgow), Rangoon Oil Company, British Burmah.  
 Donald, William (late of Saltecoats), Kennall Gunpowder Works, Kennall Vale, Penrhyn, Cornwall.  
 Duggan, T. R., 14, Alkham Row, Stoke Newington, N. (late of South Hackney).  
 Fahlberg, Constantine (late of Philadelphia), care of Caesar Wall, Esq., 243, Bowery, New York, U.S.A.  
 Gadsden, H. A. (late of Australian Avenue), 6, Crosby Square, Bishopsgate Street, London, E.C.  
 Gibb, Thomas (late of Grange Metal Works), Jarrow-upon-Tyne.  
 Gunn, W. L. (late of Sheffield), care of Palmer and Company, Victoria Oil and Candle Works, Stratford, London, E.  
 Hanrez, Prosper, 9, Rue Moris, Chaussée de Charleroi, Brussels.  
 Hartley, Joseph (late 176), 71, Clowes Street, West Gorton, Manchester.  
 Herschel, A. S., 47, Lovaine Place, Newcastle-upon-Tyne.  
 Kean, James (late 47), 37, Croft Terrace, Jarrow-upon-Tyne.  
 King, A. J. (late of Mining Lane), Fern Bank, Withington, Manchester.  
 King, J. Westall, 115, Wellington Street, Glasgow.  
 Kingzett, C. T. (late of Tottenham), Trevena, Andmstr Park, London, N.  
 Mason, A. H. (late of Liverpool), 41, Saint Jean Baptiste Street, Montreal, Canada.  
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 Petty, A. (late of Stamford Hill), Wood Lodge, Shooter's Hill, Kent.  
 Pickles, H., Prussiate Works, Droylsden, Manchester.  
 Readman, J. B. (late of Glasgow), 9, Moray Place, Edinburgh.  
 Rowlands, W. L., 3268, Chancellor Place, West Philadelphia, Pa., U.S.A.  
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 Stevenson, James, 27, St. Vincent Place, Glasgow, and The Bromfields, Largs, N.B.  
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 White, W. H. (late of Saffron Hill), Killingworth House, Killingworth, Newcastle-on-Tyne.  
 Wright, James C., 14, Waterside Street, Irvine, N.B.  
 Yglesias, M., 2, Tokenhouse Buildings, London, E.C.

#### CHANGE OF ADDRESS REQUIRED.

Irwin, J. T. (late of 261, Blackburn Road, Acerrington).

#### RESIGNATIONS.

R. V. Bethel, R. M. Morris, H. Salvin Pattinson, Alfred Payne, W. G. Whittam, H. J. Smith.

#### NAMES OF NEW MEMBERS ACCIDENTALLY OMITTED IN SUPPLEMENTARY LIST, FEBRUARY.

Cresswell, C. N., 1, Hare Court, Temple, London, E.C.  
 West, John, Barfield House, Didsbury, near Manchester.

## London Section.

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Dr. E. Frankland.	John Spiller.
Prof. Charles Graham.	G. C. Trewby.
F. Maxwell Lyte.	C. Unney.
Geo. Matthey.	J. Williams.

Local Sec.: Thos. Tyrer, Battersea, S.W.

The meetings of this Section are held on the first Monday in each month.

The following altered arrangements for papers for the remainder of the Session are notified:—

On April 7th, Election of Local Committee. Dr. Armstrong, "Preliminary Note on Certain By-products of the Pintsch Oil Gas Manufacture in Relation to the Question as to the Conditions under which Benzenes are Formed." Messrs. Giles and Shearer, "On the Estimation of Sulphurous Acid in its Compounds." Mr. W. L. Carpenter, "Further Note on the Solidification of Liquid Oils."

On May 5th, Dr. Percy Frankland, "On the Composition and Illuminating Power of Coal Gas." Mr. W. J. Dibdin, "On the Estimation of the Illuminating Power of Gas Burners, especially those of large size."

On June 3rd, Mr. W. S. Squire, "On the Processes Concerned in the Conversion of Starch into Alcohol, and their relation to Brewing and Distilling," illustrated.

CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE,  
Monday, March 3rd, 1884.

MR. DAVID HOWARD IN THE CHAIR.

## ON THE MANUFACTURE OF CUPRAMMONIUM AND ZINCAMMONIUM COMPOUNDS AND THEIR TECHNICAL ADAPTATION.

BY C. R. ALDER WRIGHT, D.S.C., F.R.S.

It has long been known that when an aqueous solution of ammonia is added to one of certain metallic salts, a precipitate is at first formed which redissolves on further addition of ammonia, forming a fluid from which in many cases definite crystallised compounds can be isolated by evaporation, or by treatment with alcohol or a large excess of concentrated ammonia solution, etc., so as to cause the compound to separate through being comparatively insoluble in the modified menstruum. These compounds usually contain the elements of the metallic salt originally used together with those of ammonia, and in some cases water (of crystallisation or constitutional). Copper and zinc belong to the class of metals the salts of which behave in this way. Various kinds of ammonia compounds of different salts of these two metals have been described by different observers, the best defined of which in the cupric series belong to the tetrammonio class  $\text{CuX}_2 \cdot 4\text{NH}_3$ , or  $[\text{Cu} \cdot 4\text{NH}_3]\text{X}_2$ —as, for example, cuprotetrammonium sulphate  $[\text{Cu} \cdot 4\text{NH}_3]\text{SO}_4$ ,  $\text{H}_2\text{O}$ , which is readily obtained in crystals by adding to a concentrated copper sulphate solution ammonia in large excess, or ammonia in less excess and alcohol. The similarly-constituted cuprotetrammonium bromate, nitrate, hyposulphate, and various other salts are similarly obtainable. Under the influence of heat these compounds lose ammonia, forming analogous but less ammoniated compounds. Thus cuprotetrammonium sulphate at near  $150^\circ\text{C}$ . loses  $2\text{NH}_3$ , and at about  $205^\circ\text{C}$ . loses another  $\text{NH}_3$ , and finally at about

$260^\circ\text{C}$ . loses the last  $\text{NH}_3$  present, thus forming in succession the series—

$[\text{Cu} \cdot 4\text{NH}_3]\text{SO}_4$ .....Cuprotetrammonium sulphate  
 $[\text{Cu} \cdot 2\text{NH}_3]\text{SO}_4$ .....Cuprodiammonium sulphate  
 $[\text{Cu} \cdot \text{NH}_3]\text{SO}_4$ .....Cupromonammonium sulphate  
 $\text{CuSO}_4$ .....Anhydrous copper sulphate

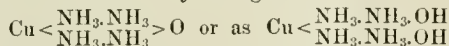
Metals the salts of which form with ammonia compounds of this kind are usually capable of giving rise to another class of ammonio-metallic derivatives by dissolving their oxides or hydroxides in excess of ammonia solution. In some cases, *e.g.*, with copper, these fluids are readily formed by agitating scraps of the metal with air and ammonia solution, the metal spontaneously oxidising under these conditions with considerable rapidity. In other cases, *e.g.*, zinc, the metal when in contact simultaneously with air and ammonia solution does not oxidise rapidly, but the action can be brought about more readily by conjoining to the chemical agencies at work the galvanic effect of the admixture of another metal in contact with the one to be dissolved. Thus a mixture of zinc and iron cuttings is far more effective in producing solution of the former metal than the cuttings of zinc alone, although the iron remains practically unaffected. It is remarkable that brass dissolves in ammonia as a whole, both zinc and copper going into solution under the influence of aerial oxidation. It is usual to assume that the compounds formed by the union of ammonia with metallic salts, oxides, or hydroxides, are the corresponding salts, oxides, or hydroxides of compound metallo-ammoniums. From this point of view cuprotetrammonium sulphate may be regarded as—



or ammonium sulphate, in which  $\text{H}_2$  are replaced by the diad group—



In the same way the solution of copper oxide in excess of ammonia may be regarded as—



Similar remarks apply to the zincammonium derivatives. In the case of cupro- and zinc- ammonium compounds, the stability of the substances under ordinary conditions is not great. Even at ordinary temperatures there is usually a more or less marked tendency to decompose evolving ammonia; thus cuprammonium hydroxide solution is decomposed by addition of a large bulk of alcohol, a blue precipitate being thrown down consisting essentially of hydrated copper oxide (usually also containing a little carbonic acid and ammonia associated therewith); and on long standing (some weeks or months) concentrated solutions deposit a black substance lining the containing vessel, essentially consisting of anhydrous copper oxide. Similarly, cuprotetrammonium sulphate crystals are broken up by contact with a large bulk of cold water into free ammonia, ammonium sulphate, and a blue precipitate of variable composition, but essentially consisting of a "basic" sulphate,  $\text{CuSO}_4 + \text{NCuO}_2\text{H}_2$ , also retaining a little ammonia. In presence of excess of ammonia, cuprotetrammonium sulphate wholly dissolves to a blue liquid, which, if strongly ammoniacal, may be evaporated down on the water-bath without any considerable amount of change, forming a thick residue mostly soluble in a small bulk of water. On continued boiling of a less ammoniacal solution, however, black cupric oxide is formed, with more or less basic sulphate. Some interesting illustrations of the influence of mass and temperature on decomposition are afforded by these reactions; into these points, however, it would be out

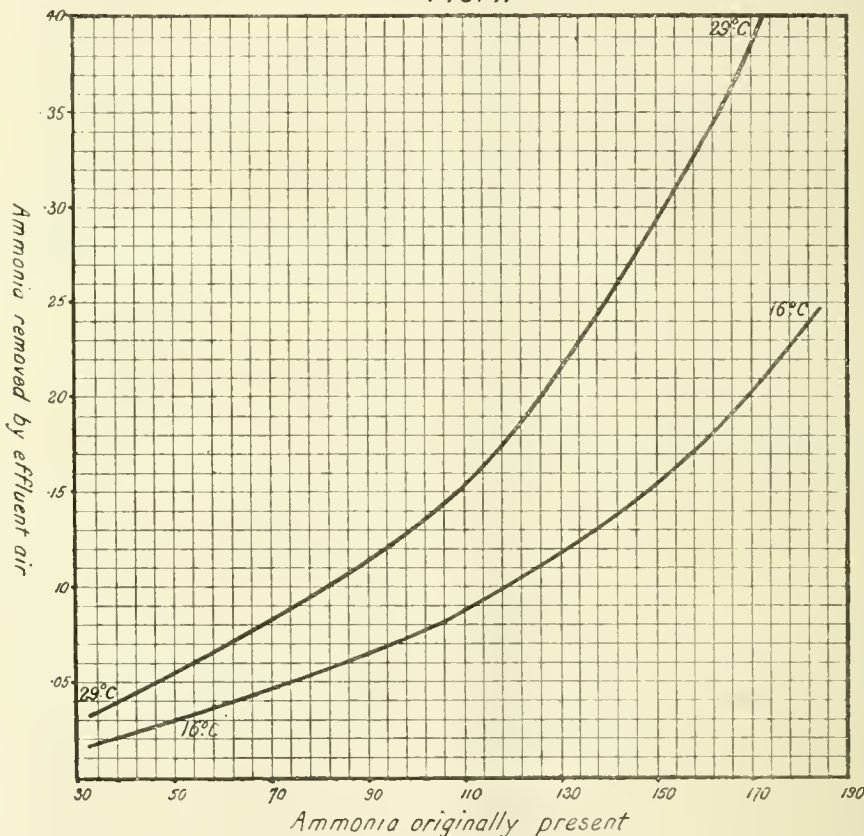


of place now to enter. It is noteworthy that zinc ammonium hydroxide solutions deposit, on long standing, crystals essentially consisting of zinc hydroxide,  $\text{ZnO}$ ,  $\text{H}_2\text{O}$ . If carbonic acid be excluded, the crystals are often perfectly clear and transparent, but are more or less white and opaque, and consist of a "basic" carbonate,  $\text{ZnCO}_3 \cdot \text{N} \cdot \text{ZnO}_2 \cdot \text{H}_2\text{O}$ , if the solution contains ammonium carbonate.

According to Wetzlar cuprammonium hydroxide solution containing sal-ammoniac or certain other salts attacks metallic iron, causing copper to be deposited in the metallic form and oxide of iron to be produced. Pure cuprammonium hydroxide containing a large excess of ammonia, however, is found in actual practice on the large manufacturing scale to cause no noticeable corrosion of either wrought or cast iron, or of steel, so that receptacles for the fluid,

considerable scale to practical manufactures, although various modes of employment of them have been from time to time proposed and even patented. For a long time these fluids have been used in small quantities as tests of the character of certain organised fibres, *e.g.*, silk, cotton, linen, etc., on account of their power of dissolving, or at any rate of pectising, gelatinising, and disintegrating\* these fibres. Recently C. F. Claus has patented the production of a kind of silicate of zinc by the addition of silicate of soda to ammoniacal zinc solution; whilst the zinc hydroxide left on goods treated with zincammonium hydroxide solution and exposed to the air is capable of acting as a mordant, although it does not appear to have been largely employed in that direction as yet. Of late years, however, an attempt has been made to utilise on a manufacturing scale the remarkable power

FIG. 1.



and also machinery, can safely be constructed of iron or steel without fear of injury, even when kept in prolonged contact with the solution. In this respect iron differs remarkably from zinc. Both metals readily displace copper from solution in ordinary salts, *e.g.*, copper sulphate; but with regard to an ammoniacal solution their behaviour is not the same, iron having no action, but zinc precipitating copper as readily as from an ordinary salt, and forming in so doing zincammonium hydroxide solution—in fact, this reaction forms a most convenient way of preparing that fluid on the large scale.

Owing probably to the want of permanence of cupro- and zinc- ammonium compounds, and the cost of the large quantity of ammonia solutions requisite to form and preserve them, these substances do not appear as yet to have been applied on any

possessed by cuprammonium solutions of pectising cellulose and similar substances, in such a fashion as to produce a partial alteration somewhat analogous to that brought about by dipping paper into sulphuric acid so as to "parchmentise" it. After much laborious experimentation, the processes originally patented by the late Dr. John Scoffern have been improved and reduced to a regular manufacture by the Patent Waterproof Paper and Canvas Company, of the Canal Works, Willesden Junction, whose specialties are known as "Willessden" fabrics, amongst which may be particularly noticed a waterproof paper for lining damp walls, a variety of thicker cards for roofing and

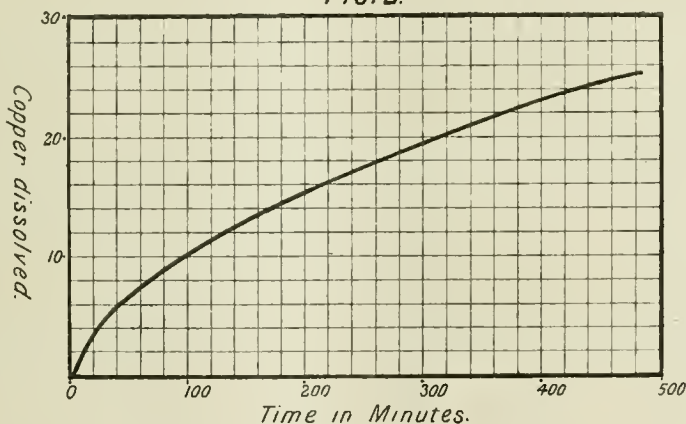
\* According to Erdmann (*J. Prakt. Chem.* lxxvi., 385) cellulose is not truly dissolved by ammoniacal copper solutions, but is only disseminated through the fluid in a gelatinised condition.

other analogous purposes, and canvas and ropes, etc., prepared so as to be rotproof.

The process by which these fabrics are manufactured may be described as essentially consisting of the preparation of a concentrated solution of cuprammonium hydroxide, and the passing of the goods to be treated through a bath of this material at just such a rate as will permit of the pectising and gelatinising of the exterior of the fibres composing the paper or canvas, etc., without wholly disintegrating the mass; so that the material on emerging from the bath retains coherence sufficient to enable it to be passed over and under the usual drums, etc., of a paper mill, and so to be dried in the ordinary way. This drying converts the film of pectised cellulose coating each filament and fibre into an insoluble solid varnish which cements the whole together, forming a material entirely *sui generis*. In order to build up thick cards two or more reels of paper are employed, passed simultaneously through the bath, and then pressed together and dried as a whole, two thicknesses thus treated forming two-ply card. The best kinds of thick cards are made by passing two rolls of two-ply simultaneously through the bath a second time and pressing them together and drying, thus giving rise to four-ply card, the thickness mostly adopted for roofing. By repeating this process with two batches of four-ply, eight-ply is obtained; and similarly to any required degree of thickness necessary for special purposes.

ever, does not pectise paper sufficiently to give good results. In order to pectise paper, etc., thoroughly when the materials are passed through the baths at a convenient manufacturing speed, it is essential to use a liquid containing 100 to 150lb. of ammonia per 100 gallons \* (about as much ammonia as is present in solution of ammonia, specific gravity '940 to '960). Such a fluid, when nearly saturated with copper present as pure cuprammonium hydroxide, will contain 20lb. to 25lb. copper (reckoned as metal) per 100 gallons. Considerably larger amounts of copper, however, may be taken into solution in the form of cuprammonium salts, or when certain forms of organic matter are also present in the liquor. According to the textbooks, ammoniacal solutions of cuprammonium salts dissolve cellulose, but such fluids are found to be unsuitable for the manufacture of Willesden goods for a variety of reasons. In the first place, for a given quantity of copper in solution an ammoniacal solution of cuprammonium hydroxide appears to possess a considerably higher pectising power than a similar solution of a cuprammonium salt; next, when a cuprammonium salt, *e.g.*, the sulphate, is used there is not only a tendency to form a little copper sulphate, which can be washed out of the finished fabric by water (thus rendering the material unsuitable for many purposes, *e.g.*, cattle drinking-troughs, portable sheep-pens, etc.), but further, much ammonium sulphate is formed in the body of the fabric, thus giving rise to a double dis-

FIG. 2.



A noteworthy point in connection with these processes is that, when certain precautions are taken, the copper present in the ammoniacal fluid imbibed by the material passed through the bath is wholly converted during drying into a compound with the pectised cellulose of an agreeable green tint, and is not deposited as black oxide of copper, as it would be on evaporation of the solution without cellulose in a porcelain dish, etc. It is largely the presence of copper in this form that renders Willesden fabrics so free from growths of mould, mildew, rot, and fungoid vegetation generally, and from the attacks of insects. The compound is so stable as to be wholly unaffected by water, when once dry. Of course, mineral acids dissolve out copper to some extent, but this is not the case with ordinary water, apparently not even with London rain. Instead of cuprammonium hydroxide alone, in certain cases a mixture of cuprammonium and zincammonium hydroxides may be used. The pectised cellulose then contains both zinc and copper, indicating apparently that a zinc cellulose compound has also been formed. Zincammonium hydroxide alone, how-

ever, does not pectise paper sufficiently to give good results. In order to pectise paper, etc., thoroughly when the materials are passed through the baths at a convenient manufacturing speed, it is essential to use a liquid containing 100 to 150lb. of ammonia per 100 gallons \* (about as much ammonia as is present in solution of ammonia, specific gravity '940 to '960). Such a fluid, when nearly saturated with copper present as pure cuprammonium hydroxide, will contain 20lb. to 25lb. copper (reckoned as metal) per 100 gallons. Considerably larger amounts of copper, however, may be taken into solution in the form of cuprammonium salts, or when certain forms of organic matter are also present in the liquor. According to the textbooks, ammoniacal solutions of cuprammonium salts dissolve cellulose, but such fluids are found to be unsuitable for the manufacture of Willesden goods for a variety of reasons. In the first place, for a given quantity of copper in solution an ammoniacal solution of cuprammonium hydroxide appears to possess a considerably higher pectising power than a similar solution of a cuprammonium salt; next, when a cuprammonium salt, *e.g.*, the sulphate, is used there is not only a tendency to form a little copper sulphate, which can be washed out of the finished fabric by water (thus rendering the material unsuitable for many purposes, *e.g.*, cattle drinking-troughs, portable sheep-pens, etc.), but further, much ammonium sulphate is formed in the body of the fabric, thus giving rise to a double dis-

\* 1 gramme per litre = 1lb. per 100 gallons, a relationship convenient to remember, when laboratory tests are made using the metrical system, whilst British weights and measures are used in the actual works.



vegetable fibres by its solvent action thereon. For the economical production of the fluid at the rate of several tons at an operation, apparatus and methods had to be devised, more especially with the twofold object in view of causing the solution of copper in as short a period as possible, and of preventing ammonia being lost in large quantities during the operation. Whilst pursuing the investigations necessary for the attainment of this object, several points of interest were observed which may be here fitly recorded.

As to the relationship between the ammoniacal strength of aqueous solution of ammonia and its specific gravity at some assigned constant temperature. A number of careful observations were made (in conjunction with Mr. C. Thompson) with different aqueous solutions of ammonia, the relative weights of equal bulks of the solutions and of distilled water at 18° C. being determined, and also the percentage of ammonia present. From the results obtained the figures given in the second column of the following table were deduced by interpolation. The third and fourth columns show respectively the percentages of ammonia found by Carius and by Otto in liquids of the gravities cited, the temperature of comparison with water being 14° C. and 16° C. respectively. The most probable mean results for the temperature 16° C., taking into consideration all the observations, are given in the fifth column, whilst the sixth column represents the number of grammes per litre (or pounds per 100 gallons) of ammonia present thence deduced, and the seventh the difference in number of pounds per gallon per '001 difference in specific gravity.

Sp. Gr.	Per cent. of Ammonia at 18° C. Alder Wright and Thompson.	Per cent. of Ammonia at 14° C. Carius.	Per cent. of Ammonia at 16° C. Otto.	Most probable Mean at 16° C.	Grammes per litre (or lbs. per 100 gallons) at 16° C.	Difference per '001 sp. gr.
1'000	0	0	—	—	0	—
'995	1'16	1'200	—	1'17	11'64	2'33
'990	2'32	2'375	—	2'31	23'17	2'31
'985	3'48	3'525	—	3'52	34'67	2'30
'980	4'74	4'725	—	4'73	46'35	2'34
'975	6'00	5'975	5'850	5'97	58'21	2'37
'970	7'27	7'225	7'175	7'23	70'13	2'38
'965	8'54	8'514	8'500	8'52	82'22	2'42
'960	9'81	9'825	9'800	9'83	94'37	2'43
'955	11'19	11'171	11'125	11'16	106'58	2'44
'950	12'56	12'513	12'416	12'53	119'04	2'49
'945	13'91	13'971	—	13'96	131'93	2'58
'940	15'47	15'100	—	15'44	145'14	2'61
'935	17'01	16'900	—	16'96	158'58	2'68
'930	18'54	18'467	—	18'50	172'05	2'69
'925	20'08	20'033	—	20'06	185'55	2'70

The following table indicates the results of some experiments on the alteration in specific gravity produced by temperature variation with liquids of various strength:—

Sp. gr. at 18° C.	Variation in sp. gr. per 1° alteration in temperature.
'925	'0005
'940	'0003
'960	'0001
'980	'00005

Hence a liquid of specific gravity '925 at the temperature 16° would at 18° become lighter by an amount substantially equal to  $(18-16) \times '0005 = '001$ , i.e., its specific gravity at 18° would be '925 - '001 = '924. Similarly at 20° its specific gravity would be '925  $(20-16) \times '0005 = '923$ . Again, a liquid of specific gravity '940 at 21° would have at 16° the specific gravity '940  $+(21-16) \times '0003 = '9415$ , and so on. These figures do not pretend to extremely minute accuracy, but they are quite accurate enough to show that a difference of two or three degrees in the tem-

perature at which the specific gravity is tested will make a very material difference in the amount of ammonia present deduced from the specific gravity determination, the effect being more marked the stronger the solution. For instance, a fluid testing '925 at 20° C., instead of containing 185'55 lb. of ammonia per 100 gallons (grammes per litre), as it should do were its specific gravity '925 at 16° C., will only contain as much ammonia as solution of specific gravity '925  $+(20-16) \times '0005 = '927$ ; that is, will only contain 185'55 -  $2 \times 2'70 = 180'15$  lb. per 100 gallons, or almost exactly 3 per cent. less ammonia. Inasmuch as temperature observations are often altogether omitted in testing goods by specific gravity, whilst thermometers, if used, are apt to be erroneous by two or three degrees, especially those of the cheaper kinds—and further, inasmuch as hydrometers are rarely exact within  $\pm '001$ , and are often erroneous to a much greater extent—it is evident that the above table of ammoniacal strengths is far more sharply exact than the exigencies of commerce require; and further, that if estimations of strength of higher accuracy than say 5 per cent. be desired, the indications of the hydrometer must not be relied upon, but a proper quantitative analysis must be made. *Mutatis mutandis*, the same remarks apply to the testing of most other fluids by means of the hydrometer. In connection with this subject it is of interest to notice that the specific gravity of a pure cuprammonium hydroxide solution is found experimentally to be always higher than that of an aqueous solution of ammonia alone, containing the same amount of ammonia and at the same temperature, by an amount equal to  $n \times '00159$  where  $n$  is the number of pounds of copper (as metal) present per 100 gallons (grammes per litre). Several comparisons with liquors containing widely-different amounts of copper and of ammonia led to numbers differing from the mean constant '00159 by quantities in no case exceeding the limits of observational error. Thus, for instance, a solution of cuprammonium hydroxide containing at 16° C. 10 lb. of copper and 119'04 lb. of ammonia per 100 gallons has the specific gravity at 16° C. of '950  $+ 10 \times '00159 = '9659$ .

As to the quantity of ammonia capable of being taken up by air in contact with aqueous ammonia solutions of various strengths at common temperatures.—At the ordinary temperature and pressure (say 15° C. and 760 mm.) 100 gallons of air contain about 255 lb. of oxygen, capable if fully absorbed, of converting rather more than 1 lb. of metallic copper into cupric oxide. Hence, in order to dissolve in 100 gallons of ammoniacal solution 25 lb. of copper, a volume of air will be requisite equal to about 25 times the bulk of the solution (2,500 gallons) if all the oxygen be removed, and proportionately more if only a portion of the oxygen be absorbed. It is obvious that the unused nitrogen or "spent air" will carry away a considerable amount of ammonia, and presumably more the higher the temperature and the stronger the ammoniacal fluid used. In actual practice it is found that the ammonia thus carried away is so large a fraction of that originally present in the ammoniacal solution as to make it indispensable to recover it. In order to determine with a fair approach to accuracy the actual amount of ammonia thus carried away in different cases, several series of observations were made with air saturated with moisture and ammonia vapour by slowly bubbling it through a bulk of ammonia solution large enough to undergo no material diminution in strength during the experiment. The ammonia was determined by passing the air through an acid solution of known strength, and the quantity of air employed by simply collecting and measuring it. The following table gives the mean results of a number

of concordant observations thus made at 16° C. and at 29° C. :—

Pounds Ammonia per 100 gallons in the fluid employed to saturate the air.	Pounds Ammonia removed per 100 gallons of effluent air measured at 0° and 760mm.		
	At 16° C.	At 29° C.	Average Difference per 1° C.
188	·249	·546	·023
164	·176	·355	·011
141	·130	·252	·0094
121	·096	·182	·0066
92	·063	·118	·0042
62	·038	·070	·0025
33	·0185	·032	·00103

The curves drawn (Fig. 1) represent these values. It is hence evident that to dissolve 25lb. of copper (requiring, as above shown, some 2,500 gallons of air) will mean the carrying away of from 5lb. to 20lb. of ammonia at spring and summer temperatures from liquid originally containing 150lb. to 160lb. per 100 gallons, even if all the oxygen be utilised; whilst if only one-fourth of the oxygen be fixed by the copper, four times as much will be removed. Practical experience in the works is in harmony with these deductions, a very considerable fraction of the ammonia originally present being removed by the air employed to oxidise and dissolve the copper. Accordingly the apparatus employed at Willesden for the manufacture of cuprammonium hydroxide solution (conveniently termed "Blue liquor") is so designed as to meet this difficulty, the ammonia being regained in accordance with Alder Wright's patent in the form of a comparatively weak cuprammonium hydroxide solution.

*As to the rate of solution of metallic copper when agitated with air and aqueous solution of ammonia.*—A number of laboratory experiments were made, using a glass cylinder filled with crumpled-up fragments of sheet copper over which ammonia solution was poured. A stream of air was then made to bubble through the whole, whereby the copper was rapidly oxidised and dissolved. Samples being drawn from time to time and analysed, data were obtained for drawing curves, the times since commencing being abscissæ and the amount of copper taken into solution ordinates. Fig. 2 illustrates such a curve obtained with a cylinder containing a column of copper fragments 9 inches in height, and solution of ammonia of about 155lb. per 100 gallons. The air stream was such as to give about 40 times the bulk of fluid used per hour (measured at the ordinary temperature and pressure).

Time since commencement. Minutes.	Copper in pounds per 100 gallons of fluid.	Gain per hour.
5	1·05	—
60	7·50	7·04
150	12·15	3·30
200	15·00	3·06
215	17·20	3·13
305	19·50	2·30
365	21·15	1·95
485	25·10	1·82

It is here obvious, first, that the rate of solution gradually slackened as the liquid became richer in copper; and secondly, that even at the period when the rate of solution was most rapid, only about a sixth of the oxygen in the air used was fixed by the copper; for an air stream of 40 volumes per hour would suffice to dissolve slightly more than 40lb. of copper per hour per 100 gallons, whilst 7·04 was the maximum rate actually observed. Precisely analogous results were obtained in numerous other experiments with copper columns of very varying magnitudes; *ceteris paribus* increasing the length of column increased the rate of

solution. But even with a column 30ft. long the majority of the oxygen of the air escaped unabsorbed except when this rate of passage was so slow that it would necessarily introduce practical inconveniences through delay in manufacture if such a method were adopted. Based upon the knowledge derived from the foregoing and other analogous laboratory experiments, a system of manufacture of blue liquor was finally devised, which, after trying on a small manufacturing scale, and undergoing some slight modifications in consequence, was finally adopted, the result of which is that cuprammonium hydroxide solution can be manufactured without any practically appreciable loss of ammonia of any acquired richness in either copper or ammonia compatible with the degrees of solubility of those substances. On account of their spontaneous decomposition with separation of black oxide of copper on long keeping, solutions of cuprammonium hydroxide are not adapted for export or long storage, when of considerable richness of copper—at least they necessarily deteriorate as regards the amount of copper in solution, although not appreciably in any other respect. When, however, the amount of copper does not exceed some 12lb. or 15lb. per 100 gallons, little or no depreciation takes place even on keeping for a twelvemonth, whilst weaker solutions may be kept unchanged for an indefinitely long period. Such solutions appear amongst other purposes to be well adapted for the treatment of sleepers and timber generally for the purpose of preservation against decay and insect life. Zincammonium-hydroxide solutions also appear to possess properties rendering them susceptible of various useful applications. They are readily obtainable by means of the methods used for the preparation of cuprammonium hydroxide. Copper scrap, or preferably refuse brass, is dissolved up in ammonia, and the mixed copper and zinc solution obtained digested with old zinc (worn-out roofings, gutters, etc.), so as to throw down the copper as precipitated copper, which may be dissolved up over again if required, and form a solution containing zinc only. Besides the interest to chemists afforded by the peculiar properties of these ammonio-metallic solutions, and to chemical manufacturers by the introduction of a method of manufacturing them in quantity without considerable expense, there is another point connected with the matter which may be of some interest to the Society of Chemical Industry, viz., the various directions in which the Willesden fabrics, and more especially the card, can be turned to account in a factory. For the construction of light yet extremely durable roofs and sheds either permanent or portable; for temporary offices and workmen's tents used during the construction of large engineering works, *e.g.*, bridges, railways, etc.; for shelters to keep rain off hay and corn ricks, barns, and numerous other farmyard purposes; for drinking troughs, water-storage tanks, sheep-pens, portable photographer's dark rooms, sentry boxes, bathing boxes, boathouses, and boats; for the construction of gutters and pipes apparently unalterable either above or below ground, even after several years' service; and for a number of other similar purposes the material has already been tried with most satisfactory results—for instance, a large quantity of this material was used for roofing and shedding purposes at the Fisheries Exhibition, where its rain and weather resisting qualities were so well tested as to gain the award of a silver medal. The thinner materials, one-ply and two-ply, are well adapted to the lining of packing cases to keep out moisture, and to wall papering of damp rooms, as an under coat preventing moisture creeping through; whilst waterproof envelopes and stationery and light sample cases and parcel post packages are amongst the other directions in



which this material finds useful application. Canvas and cordage treated by the Willesden process are not increased in weight or rigidity, as is the case when tarred and otherwise treated; whilst a high degree of freedom from mildew, mould, rot, and decay, and greater durability generally are thus secured.

A fuller description of the various uses to which these Willesden fabrics may be put will be shortly given in a paper on the subject to be communicated to the Society of Arts.

#### DISCUSSION.

The CHAIRMAN said he was sure the Society would thank Dr. Wright for bringing before them a very useful application of a very well-known reaction. It was very evident that such a method of waterproofing fabrics and rendering them indestructible must have many practical applications. It was interesting to know from recent experiments made in France with regard to the most effectual method of destroying poisonous germs, that in many cases sulphate of copper exceeded all other disinfectants. Of course in moist climates, where mildew and rot were specially to be feared, these substances treated in the manner described would be practically indestructible. Sleepers had been already pickled with sulphate of copper to a certain extent, but the addition of the ammonium salt with its solvent action on the cellulose, pointed to the possibility of a much wider application of this process.

Mr. TYLER asked whether the thick ropes, samples of which were shown, were waterproofed throughout, or was the exterior only waterproofed.

Dr. WRIGHT replied that the process as applied to ropes and canvas was not so much intended to render them waterproof in the sense of keeping the rain out, as to prevent mildew and rot, to which ordinary canvas was subject. He might call attention to the various specimens exhibited, showing the different effects on strips of canvas, paper, and card, of immersion in water or moist air when treated with this liquid and when left untreated. If the materials were boiled the effect would be still more marked; treated canvas might be boiled for any length of time without any apparent effect of any kind being produced, whilst ordinary canvas would be more or less injured, and of course paper or cardboard would speedily go to pulp. There was another specimen in which several strips of paper had been dipped in the solution for a portion of their length and then passed through rollers, when they became inseparably agglutinated together, and would sustain a much greater weight than before.

Mr. JOHNSON asked if the process added anything to the strength of the card.

Dr. WRIGHT said the amount of strain the material would bear after treatment was very greatly increased, as could be proved by attaching a weight to it.

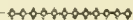
Mr. NEWLANDS suggested that the same effect would be produced by merely gumming the sheets together. He should like to know if this process was applicable to filtering cloth, and if it would strengthen it. If so, even at a considerable cost, there would be a considerable use for it. Scrim cloth was mentioned, and that was used largely in the sugar manufacture, but it rapidly wore out.

Dr. WRIGHT remarked that there was a great difference between a material formed of several sheets of paper by this process and a piece of pasteboard made in the ordinary manner. The latter (the pasteboard) would be reduced to its pristine elements by exposure to water; but Willesden card would not be affected, the agglutination being permanent. With reference to the possible superiority of the prepared canvas as a filtering material, he was not aware that

it had ever been tried; but he should imagine it would be somewhat less porous than before, on account of the filters being coated with a kind of glutinous cement, which would render the crevices between the warp and woof smaller. But if a somewhat more loosely-woven canvas were treated, it would then no doubt be as permeable to fluids as a more closely-woven kind untreated; and no doubt it would resist the action of boiling saccharine fluids, which he supposed Mr. Newlands referred to.

Mr. EAST asked if it would be possible to treat the material for canvas and ropes before manufacture. That would get over the difficulty just referred to.

Dr. WRIGHT said there was no doubt the hemp fibres might be dipped, but he did not think they would spin so well. If that could be got over by altering the machinery there would be no other obstacle. He should fancy the fibres would be more like bristle after dipping.



#### ON THE FILTRATION OF POTABLE WATER.

BY S. H. JOHNSON.

THERE are few subjects of greater importance to the community in general than the quality of water used for drinking purposes. With the increase of civilisation and the consequent aggregation of the populations into cities and towns, the difficulties have ever increased, in consequence of the pollution of all the readily accessible water by the excreta and refuse of the people. In examining into the subject of water supply it is most striking that, from age to age up to the present, great ignorance as to the transmission of disease by polluted water should be almost universally prevalent, and we do not find until within the present century that our knowledge of the subject has received any important acquisitions. The reason of this is clearly due to the absence of chemical knowledge and ignorance of the inductive methods of research, and although our knowledge of the subject has been greatly expanded during the past fifty years, yet it must be admitted that at present we are only on the threshold of that knowledge which the importance of the subject merits and demands. Intimately connected with the subject of water as a beverage is the zymotic theory of disease, and although the specific organisms producing cholera, diarrhoea, or typhoid symptoms have not been identified, yet the evidences by analogies of results produced, and the similarity of effects of various reagents and conditions on infected liquids are so marked, when compared with like treatment of known organisms, that the conclusion that they are vital organisms, not capable of transmutation, is almost irresistible. However, whether this be so or not, there can be no doubt whatever that the specific poisons producing such diseases are in the vast majority of cases traceable to, and derived from, organic matter in a state of decomposition, and in nearly every instance such organic disease-producing matter has proved to be derived from infected human excreta in sewage, contaminating the drinking water of the persons attacked by the disease named. It is not the object of this paper to do anything further than state these views, about which so much already has been written. It may be taken as granted that the general consensus of scientific opinion is in favour of the idea of the propagation of these diseases by living organisms, and that these organisms are derived from drinking contaminated water. It is well we should have a few striking examples of the evils resulting from drinking contaminated water fresh in our memory before proceeding with the subject of filtration.

**CASE I.\***—In the year 1866 an epidemic of cholera visited London, from which the mortality was, in the eastern district, 3,700, and in the rest of London 660. It has been demonstrated to have arisen in this way: A sailor was landed at Southampton suffering from Asiatic cholera, who went on to London, and it is established that the bowel discharges of this patient passed into the Lea at a point 200 feet distant from one of the service reservoirs of the East London Water Company at Old Ford. There is also conclusive evidence that on several occasions, about this time, this infected water was admitted into the service reservoir without filtration, the supply of filtered water having run short, and this water was distributed by the East London Water Company, and it was found that "the area of intense cholera in 1866 was almost exactly the area of this particular water supply, nearly, if not absolutely, filling it, and scarcely, if at all, reaching beyond it."

**CASE II.**—A district of London on the south side of the river was visited with special severity by Asiatic cholera as often as it had been epidemic in London. On examination it was found that this district was co-extensive and continuous with the district supplied by two of the London water companies who drew their supplies from the grossly-polluted water of the Thames at Battersea—the Lambeth and the Southwark and Vauxhall Companies. These were competing companies, and a duplicate system of mains side by side supplied the houses with water indiscriminately, and in the same streets, in fairly even proportions. In the visitation of cholera of 1848-9 the tenants of each of the companies suffered in about equal proportions. In the next outbreak of cholera, 1853-4, the source of supply for the water had been changed from Battersea to Thames Ditton by the Lambeth Company, and the Southwark and Vauxhall Company continued to draw their supply from the grossly-polluted water of the Thames at Battersea. Here, then, we had an intermingled population, part of whom were supplied with a somewhat diluted and strained sewage, compared with which the remainder might be said to have been supplied with fairly pure water. That portion of the population supplied with foul water had a death-rate from cholera of 130 to every 10,000, and the remainder, who drank the moderately polluted water, had a death-rate from the same cause of 37 to every 10,000, so that the population drinking foul water "suffered  $3\frac{1}{2}$  times as much mortality as the population drinking other water," and there was an analogous correspondence of the deaths from diarrhoea. The number of lives involved in these experiments was 511,000, so that the results obtained are most reliable and conclusive, the two portions of the population being alike in all other conditions with the exception of water supply.

**CASE III.**—The violent outbreak of cholera which took place in the year 1854 in a small district in Golden Square, St. James's, Westminster, by which, within a radius of three minutes' walk of a polluted well, 609 persons lost their lives in the course of a few days. "It was shown by Dr. Snow (1) That the outbreak, properly so called, was principally confined to the area about the Broad Street pump. (2) That 61 out of 73 persons who died during the first two days had been accustomed to drink the pump water either constantly or occasionally. (3) That the water was used in various other ways, and might so have been taken in cases where its use in the ordinary way could not be distinctly traced. (4) That in the workhouse, where the well water was not used, only

5 deaths occurred, whereas 50 would have been a ratio proportionate to that of the neighbourhood around. (5) That in a factory employing 200 people, where the water was drunk daily, 18 people died. (6) That 70 men employed in the brewery in Broad Street never drank the water, and escaped cholera. (7) That in a number of individual instances, which was particularly investigated, the drinking of the water was followed by cholera. In one case a lady living quite away from the district, who had the water sent out to her, died after drinking it. Her niece also died under the same circumstances. (8) That at any point decidedly nearer to another pump the mortality from cholera as a rule ceased, and that in an inquiry extending over 48 fatal attacks which took place nearer to another pump, many apparent exceptions were found to be cases of death in persons who really had a preference for the more distant Broad Street water. (9) That in a particular street, containing 14 houses, the only four which escaped without a death were those in which the Broad Street water was never drunk. (10) That this water was used for drinking purposes only, and was used cold. From all these several facts Dr. Snow is of opinion that the outbreak was attributable to the well water as the medium of dissemination of the cholera poison. He believes, moreover, that the well water must have been not merely generally contaminated by cesspool drainage, but especially with the evacuation of a cholera patient. These conclusions of Dr. Snow have been confirmed in a remarkable manner by Mr. Whitehead, who at first entertained adverse views and investigated the matter independently.

**CASE IV.**—The great unhealthiness of Millbank Prison was for a number of years a source of great anxiety, and many measures were resorted to for its improvement, but in vain. Typhoid fever was especially fatal. At length, in the year 1854, the supply of the prison with water pumped from the Thames was discontinued, and the water raised from the artesian well in Trafalgar Square was substituted for it. Immediately on the introduction of the new supply the health of the prisoners improved, and from that date up to 1872, a period of 18 years, there have been only three deaths from typhoid fever, not one of which was contracted in the prison. Although at previous times the prison was so unhealthy that at one time as many as 500 convicts out of 880 were sick in 1849, and the death rate had reached 82 per 1,000, the average since the Thames water has been replaced does not much exceed 12 per 1,000.

**CASE V.**—At Caterham and Redhill a well-authenticated instance of the pollution of the water supply in a group of towns and villages of the specific poison or organism of typhoid or enteric fever was brought before the Society of Arts in the year 1879, by Dr. Thorne, at the Conference on National Water Supply, Sewage, and Health. It appears that an outbreak of enteric fever occurred simultaneously in this cluster of towns and villages, and 196 persons were attacked within 14 days, 21 of whom died. Since some of the affected districts were quite isolated from the rest, the simultaneous character of the attack in each of the places was very remarkable. This led to an investigation of all the usual conditions likely to affect the case. The sewage and drainage, the means of disposal of excreta and refuse, the milk supply, etc., were each inquired into, but nothing could be found throwing any light on the subject; but, on the contrary, in these matters, there was no condition in common in all the places affected. The single prevailing condition in common in all the places was the water supply; but this was thought to be almost above suspicion, being derived from wells more than 500ft. deep in the chalk. However, in the

\* The cases Nos. I. to IV. are abbreviated from the Sixth Report of the Royal Commissioners (1868) on Preventing the Pollution of Rivers.



infected district there were 1,400 houses which were not supplied by the water from the company's mains, and it was found that not one person was attacked who were occupants of these houses, who had not drunk of the water from the company's mains. This was almost conclusive that it was in the water supply the mystery would be solved. An investigation was made, and it was found that the water company had been constructing an adit between two of their wells at this time, in which a workman was employed who, it was afterwards discovered, was suffering from an attack of enteric fever, and it was also proved, and admitted by this man, that, notwithstanding the regulations to the contrary, he was compelled to evacuate the diarrhoeal discharges in the adit. The symptoms in the case were well established as those of enteric fever. This quite explained the mystery. There are literally thousands of cases which could be adduced to show that a fruitful source of prevalent zymotic diseases is the drinking of water polluted with organic matter of sewage—indeed, it has been computed that the deaths from this cause alone amount to at least 120,000 annually in the United Kingdom! So that it may be said with truth that England has lost far more by drinking dirty water than by the deaths of all the soldiers and sailors that have fallen in all the battles she has ever fought, and yet England in sanitary matters, and especially in the matter of sewage disposal, is far in advance of every other nation. From the cases heretofore cited, it is evident that when potable water acts as a carrier of infectious diseases, the infection is either derived from, or always associated with, organic matters of animal origin. It does not, however, follow that all animal organic matter contained in water must necessarily be morbid in its effects—rather the contrary is proved by all our experience hitherto. The very fact of the occurrence of epidemics of particular types of disease shows clearly that the existence of infection in a water is not the invariable result of animal contamination, but rather the exceptional condition. In the absence of any known method of determining the presence of infection in water, it is considered that the only safe course is the rejection of such as have an appreciable amount of organic pollution of animal origin. The zymotic theory certainly appears to explain the facts connected with the occurrence and transmission of the class of diseases we have been considering in a way that no chemical theory, when compared with it, can for a moment appear tenable. The assumption that living organisms of intransmutable type and of exceedingly minute dimensions are the cause is being daily more and more strengthened by the researches of biologists like M. Pasteur and others, and the identification of the specific disease-producing ferments and their habits, such, for instance, as that producing splenic fever in sheep, add confirmation to this view. On this assumption the necessity for the presence of certain kinds of organic matter is apparent, as they doubtless form a suitable pabulum for their growth and development, existing like bacteria and other ferments, by breaking down colloid organic matter of complex molecular structure into simpler forms or else into inorganic bodies.

Having now generally stated the grounds for objection to the use of water polluted with sewage for drinking purposes, apart from any sentimental objections, there can be no doubt that the subject is of vital interest to every individual to whom only a supply of more or less polluted water is accessible. With the water supply of our cities and large towns in many cases no exception can be taken. The cities of Liverpool, Manchester, Glasgow, Dublin, and Edinburgh are supplied with upland

waters above the region of any sewage contamination, but London, Birmingham, Bristol, and a large proportion of the smaller towns of the United Kingdom are still supplied with sewage-contaminated water from rivers and surface wells. In London, excluding the district supplied by the Kent Water Company, the average quantity of sewage contamination in the water supplied for drinking purposes is 3 per cent., or about one quarter of a pint of sewage per gallon, an amount that is not pleasant to realise. There is no doubt that the proper remedy is to change the sources of supply from the polluted waters of the Thames and the Lea either to wells in the chalk, the lakes of Cumberland, or the head waters of the Severn basin; but so long as the health and mortality of the people are permitted to be subservient to the interests of capital, it is probable that even in London we shall be obliged to drink water polluted with human excreta. Although in many things the institutions and material welfare of this vast metropolis compare favourably with the best of all that can be found in past history, yet in this matter of water supply we can learn a lesson from ancient Rome—the enormous works which she constructed, the remains of which to this day form a monument to her energy and her just appreciation of the best interests of her people. If the old Romans were here, with all our modern appliances and advantages, this matter of a pure water supply would not have been allowed to linger on account of vested interests, but they would have smiled and done the work. In the paper read by my friend Mr. Hutchinson, on the last occasion of our meeting, he brought before your notice the method by which not only the whole of the solids held in suspension in the sewage of towns can be cheaply and completely got rid of without nuisance, but also a large proportion of the most objectionable colloid organic bodies, by means of precipitation with alumina and lime; and there can be no doubt but that the quality of the London water supply would be greatly improved if the excreta and refuse of all the populations, amounting to 800,000, in the upper Thames basin above the intakes of the metropolitan water companies, were clarified in this way. There would still, however, remain considerable pollution by the surface drainage from highly-manured agricultural lands. So long as we derive our drinking water from sewage-polluted rivers, the method of filtration employed at our waterworks, in the interests of public health, should be the most perfect that our present resources and knowledge can supply. How far the present means employed are adequate we shall now consider. The means employed by water companies for the purification of foul and turbid river water are of the very simplest character, and have undergone no improvement whatever since sand filtration was first recommended by Mr. Telford and Dr. Brand in the year 1828. The water is first collected into reservoirs, where the heavier impurities are allowed to subside, and then, after some days or weeks, the partially clarified water is passed through a graduated filter bed (about 6 to 8 feet deep) composed generally of successive layers of boulders, large stones, small stones, coarse gravel, fine gravel, and lastly one or two grades of sand. Through such a filter bed the water is permitted to pass at the rate of from 3 to 9 inches in depth per hour—that is to say, from  $1\frac{1}{2}$  to  $4\frac{1}{2}$  gallons per square foot per hour. For Thames water the latter speed is considered much too fast, because it is found that if the rate much exceeds 5 in. per hour some visible suspended solids pass through the filter. Thus an acre of the filter beds so prepared is capable of filtering from 60,000 to 100,000 gallons of water per hour. It has frequently been asserted

that a considerable proportion of organic matter in solution is oxidised and converted into nitrates and nitrites, but that this is not the case is evident by comparison of the water of the London companies after subsidence and before filtration and after filtration through the filter beds.\*

Names of Companies.	Nitrogen as Nitrates and Nitrites. Parts per 100,000.	
	After Subsidence but before Filtration.	After Filtration.
West Middlesex .....	229	335
Grand Junction .....	356	345
Southwark and Vauxhall .....	348	315
Lambeth .....	348	361
Chelsea .....	312	307
New River .....	310	310
East London .....	311	311
Average of 7 .....	330	327

From this table it appears that the average result of filtration of the seven companies' water who use filter beds in London shows that the oxidising effect is practically *nil*, the water having, if anything, less nitrates and nitrites after filtration. From these figures it is evident that there is nothing in the nature of surface oxidation takes place by reason of the water being brought in contact with air in the pores of the filter. It is manifest that the removal of organic matter is simply mechanical, and that the organic matter removed was simply in suspension. It requires but little consideration that one may be convinced that a sand filter is at best a rude contrivance to employ for so delicate an operation as that of filtering the excessively fine matter in suspension at times in river water; and were the case dependent on and governed by the size of the pores or interspaces between the particles of sand alone, such a filter would be altogether inadmissible. But when it is considered that the difference in the dimensions of the particles of sand and the invisible particles which give to water an opalescence is very great, and that in fact the interspaces in the sand filter are reservoirs in which the fine particles of solid matter can subside, then it is more simple to comprehend its operation. It has been found that water with a turbidity from very fine suspended matter cannot in any reasonable time be clarified sufficiently by simple subsidence in a waterworks reservoir, and notwithstanding this it can be clarified quickly by a sand filter. On the assumption that the upper surface of each grain of sand becomes a catchment shelf for the suspended matter deposited on it, the *modus operandi* is clear. We may to this end consider a filter bed as a modified subsidence reservoir having two altered conditions—namely, a greatly-extended area, and a greatly-diminished depth. Now, if any of you will take the trouble to observe, if you have not already observed the fact, you will find that the time required for matter in suspension in a fluid to completely subside is in the direct ratio of the depth of the vessel—that is, the deeper the vessel the longer time it will require. For the sake of expressing the case numerically we will assume that we have a filter bed of three acres area, the sand in which is 50in. deep, and that the interspaces between the grains of sand average 0.1in. in

depth; that there is an average of 20 such spaces to the inch depth of the sand of the filter bed; that the area of the available catchment shelves on the upper surfaces of the grains of sand in each of these twentieths of an inch is one-third of the area of the upper surface of the filter bed. Then we have—

$$\frac{20 \times 50 \times 3}{3} = 1000$$

acres, equal net area of catchment shelves, so that the filter bed is equivalent to a subsidence tank of 1,000 acres in extent,  $\frac{1}{1000}$ th of an inch deep. If we now compare this with a subsidence tank 100in. deep, and bearing in mind the fact that the time required for subsidence of suspended particles is proportional to the depth, the matter would be deposited completely in the filter bed in  $\frac{1}{10000}$ th of the time required for deposition in the subsidence tank 100in. deep. It is simple thus to understand the action of a sand filter in removing a slight turbidity in water. And it will now be readily understood how that a very slight increase in the rate of flow of the water through a sand filter will be sufficient to carry the deposited matter through the filter. Thus the work done on a given area of filter bed is of necessity limited, and hence the necessity for large areas of sand filters. If we consider the great importance of this subject of the filtration of water at our waterworks, and that it is in the solid matters in suspension that all the danger exists (for we cannot conceive it possible that the disease-producing organisms can exist absolutely in the liquid state), it would seem more desirable, if a positive filter of so fine a porous texture were used, that it would hold back mechanically all insoluble organic matter, however fine. The Porter-Clark process for softening water, which Mr. Porter so amply brought before the notice of the Society at the last meeting, much more closely approximates to this condition than a sand filter, by making use of the fine precipitate of carbonate of lime, after the softening is effected by Clark's process, as a filtering medium for the water. This is evident by the disappearance of the yellow tint due to organic matter, and present in all the London waters, and by the beautiful blue of pure water which then becomes apparent. If this process of Mr. Porter's and Dr. Clark's were substituted for the present crude and imperfect subsidence tank and filter bed, there can be but little doubt that not only would the water supply of London, or any other large town where it is required, be greatly benefitted, but it is very probable that a positive economy would result to the water companies. Although a supply of better and purer water is much to be desired, yet that alone is not sufficient protection, and so long as the present system of house cistern and intermittent supply exists additional safeguards should be employed; and in country places and villages, and towns where the water is supplied from surface wells, a good method of purifying drinking water for household use is much to be desired. If all the cases of sickness, and the maladies of various kinds which are directly traceable to the contamination of water in household cisterns, could be recorded for a single year, there is little doubt that a revelation of sickness and death from this cause would be produced only a little less astounding than that arising from poisoning with sewer gases. There are but few individuals who cannot refer to well-authenticated instances where illness affecting a household has been traced to the water cistern. It may be that the decomposed remains of a dead bird, mouse, or rat is found in the cistern, and that the mysterious illness disappears after the cleansing which then ensues. Even decaying vegetable matter, such as leaves, or the successive

\* The data for this table are taken from the 6th Report of the Commissioners appointed to Inquire into the Pollution of Rivers.



generations of vegetable growths which occur in some water mains and cisterns, are not unfrequently the source of distressing maladies, and although the necessity for scrupulous precautions to secure a pure water supply from the household cistern has been urged from time to time, yet, like many other things which are not apparent at first sight, do not obtrude themselves on our senses, no attention is paid to the subject. Things which can be seen, or discovered by the palate, at once arouse suspicion and vigorous action. Many of us would be up in arms if we found our milk watered, or our coffee mixed with chicory; but from a sanitary point of view these are venial matters compared with impure water. Daily we are in the habit of straining at the gnat and swallowing the camel in sanitary matters, and not the least in this matter of household water.

It is our object now to consider generally the various methods hitherto used for filtering water for drinking purposes in the household, and in a very brief way to examine how far they meet the requirements. All existing types of water filters may be classed under two heads: (1) *Mechanical filters*, which separate mechanically suspended solid matter from water. (2) *Chemical filters*, which have the property of removing certain bodies, such as albumen and some salts, in a more or less perfect state of solution, in consequence of a specific attraction which some bodies possess, depending on their molecular constitution, or, chemically speaking, on the particular kind of matter which forms the filter, such action not depending on the size of the pores. Mechanical filters remove the solids, because the pores are too small to permit anything but liquid to pass, and they have hitherto been made of textile or felted fabrics of every description, the pores of which are fine enough; coarse pottery or earthenware in the biscuit or unglazed state; sandstone, in various forms, particularly that called dripstone; carbon diaphragms or blocks formed of powdered coke, cemented together by means of carbon deposited by heat from sugar or tar in closed moulds; wood in thin sheets; leather; layers of finely powdered substances, such as glass, sponge, and, perhaps most effectual of all, paper. It is not necessary to enlarge on this class of filters. Chemical filters for animal charcoal and spongy iron are the principal chemical filters employed.

The annexed analytical tables, showing the effect on the organic impurities by employing animal charcoal and spongy iron respectively, have been taken from the 6th Report of the Rivers Pollution Commissioners. It will be seen that the organic constituents of water, even that which has already been filtered through a sand filter, undergoes a further very considerable reduction with both spongy iron and animal charcoal. The spongy iron is used in porous blocks, and the animal charcoal in coarse grains, and the water is passed through a small vessel filled with it. The great defects of these filters are that (1) they are bad mechanical filters, and (2) the water has to pass through the accumulated impurities of many months, and in the case of animal charcoal, owing to the presence of phosphates in large quantity (animal charcoal consisting of charcoal 10, phosphates, etc., 90), it is very favourable to the growth of low forms of vegetation and afterwards animal life. In the 6th Report the Commissioners state: "Indeed, we found that myriads of minute worms were developed in the animal charcoal and passed out with the water, when these filters were used for Thames water, and when the charcoal was not renewed at sufficiently short intervals. The property which animal charcoal possesses in a high degree of favouring the growth of the low forms of organic life is a serious drawback to its use as a filtering medium for potable waters." It has been found in

consequence of the accumulated impurities that when water is passed continuously for a long period through such porous masses, that at length the water will actually be further polluted in passing through the filter.

The essential requirements of a good household water filter are: (1) It must be a perfect mechanical filter. (2) It must be an active chemical filter. (3) It must be easily accessible for examination and cleaning. (4) It should have sufficient filtering area to supply the necessary quantity of water without delay or inconvenience. (5) The filtering medium should be replaceable with great facility, so that there should be no excuse for neglecting this precaution. (6) The arrangement should be such that nothing but pure water could be conveniently used, either for cooking or for drinking. The author having these objects in view, has endeavoured to meet all the conditions in the following way: the filtering medium

COMPOSITION OF WATER BEFORE AND AFTER FILTRATION (IN PARTS PER 100,000).

ANIMAL CHARCOAL.

DESCRIPTION.	Organic Carbon.	Organic Nitrogen.	Ammonia.	Nitrogen as Nitrates and Nitrites.	Chlorine.	Hardness.
Grand Junction Company's Water, Unfiltered	·129	·023	0	·183	1·60	19·1
Filtered.....	·029	·007	·013	·191	1·60	15·2
Grand Junction Company's Water, Unfiltered	·164	·030	·002	·062	1·90	19·7
Filtered.....	·010	·002	·002	·125	1·90	19·1

SPONGY IRON.

DESCRIPTION.	Organic Carbon.	Organic Nitrogen.	Ammonia.	Nitrogen as Nitrates and Nitrites.	Chlorine.	Hardness.
Before filtration.....	·122	·015	0	·152	1·90	20·3
After filtration.....	·028	—	·070	0	1·90	8·9
Before filtration.....	·230	·017	0	·235	2·20	21·5
After filtration.....	·060	·008	·008	0	2·20	11·8

employed consists of thick sheets of filtering paper, the peculiarity of which is that, instead of being made of ordinary paper pulp alone, a quantity of pure animal charcoal, freed from phosphates by washing with acid, is added to the pulp before it is formed into sheets. From 10 to 20 per cent. of this paper consists of purified animal charcoal. This special paper is used in a small filter press, worked by means of the pressure in the main service pipe, either in the basement of the house where the water comes in, in case of constant service, or else in the service pipe from the supply cistern in the case of intermittent service. By an inspection of the specimen filter shown, the arrangement of the filter will be readily understood. The filter press employed for this purpose, and which is shown as mounted for use, consists of a series of grooved plates on which the diaphragms are supported. The grooves in the filter plates forming the drainage surfaces, each plate has both surfaces grooved or channelled, and provided with the prepared carbon paper. These plates, with their filtering papers *in situ*, are kept apart from each other by an open frame or distance ring, and thus a battery of

filtering cells is made up, when the series of alternating plates and frames is screwed up between the fixed and loose heads of the machine. Passages for inlet and outlet are formed in the metal of the plates and frames, and suitable ports are formed so that the supply ports shall be in the distance frames conducting the water to be filtered between the paper linings of the filtering cells, and outlet ports conduct the filtered water into the common outlet channel from the grooved plates. The method of forming the internal channels in a pressure filter was originally devised by Danek. Fig. 1 shows the filter mounted against a wall filtering all the water in a service main. Fig. 2

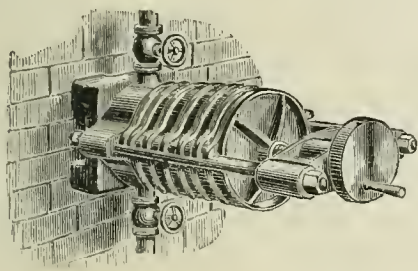


FIG. 1.

shows the filter mounted vertically, where it may be more convenient to draw off in front in that position. When it is desired to change the carbon papers for fresh ones, the filter can be shunt off on both sides from the service pipe by means of the two valves, the main tightening screw can be loosed, then all the papers become free to be removed and replaced, which is only the work of a minute or two. When screwed up the machine is again ready for work. We thus have (1) a good mechanical filter of paper with the pores filled in with fine carbon; (2) a good chemical filter with the carbon in it ten times more concentrated and

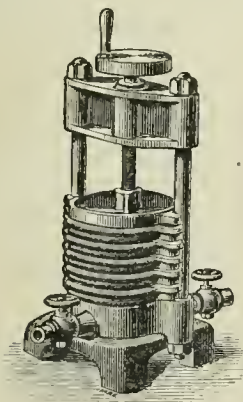


FIG. 2.

more active than ordinary animal charcoal in grains, in consequence of its being deprived of the phosphate of lime, and by the absence of which it is no longer capable of favouring the growth of the animal and vegetable organisms, which are the chief danger of the phosphatic animal charcoal in grains. (3) Nothing can be more simply accessible for cleaning, all the parts being released by means of a single screw. (4) The filtering area, even in a small apparatus, is very considerable. A machine with 6 plates has 12 filtering papers, and these working under the pressure of the main admit of the water being drawn off as required, without the necessity of the objectionable practice of keeping a store of filtered water. (5) The filtering medium in the form of these thick

paper discs can be changed with the greatest facility. By this means passing the water to be filtered through the accumulated impurities of a long period is easily avoided. No doubt, the masses will have to be educated to a knowledge of the impurity of the water they drink, and the vast amount of disease and suffering in consequence, and then it will not be thought superfluous to pay daily attention to secure clean drinking water. I had hoped to be able to give you some practical results of the working of this system, and the analytical results of the water before and after filtration, but it has not been found possible to accomplish this. But with the permission of the Society I may have an opportunity of making a short communication at a future time on this subject.

## DISCUSSION.

The CHAIRMAN said they had to thank Mr. Johnson for bringing forward an excellent method of applying the filter press to drinking water, but with regard to the general question one must remember, in justice to the much-abused water companies, that the health of London did not seem to suffer from the river water, in fact, it might be open to question whether sufficient area, especially with the presence of vegetable growth, did not afford as much safety from serious pollution as wells—at any rate unless they were extremely deep; and judging from a case which had occupied the law courts recently there seemed to be two sides even to the deep-well question. Whatever the water supply, it was necessary that it should be filtered for the purpose of drinking, and there was in this system a very perfect means of doing so, perhaps not so perfect as the combined chemical mechanical action of the Porter-Clark process, but one infinitely superior to the average of filters, some of which he had examined from time to time, and found they did not require a more delicate test than the nose to discover that the water was much better before it went in than after.

Sir FREDERICK BRAMWELL said it might interest the meeting to know, with regard to the case referred to in the paper of the pollution of water at Caterham, that there was one convincing proof of the theory advanced, and that was this: a portion of the district supplied by the Caterham company nominally was free from the disease, and it was asked how could that be if the disease was due to the water, but it turned out that that particular portion of the district was for a time supplied by another company, and, so far as the mains were supplied by that other company, there was no disease, whilst those supplied from the well in which that wretched man committed the filthy act mentioned the disease was rampant. That it must have arisen from the water was also clear from another fact. A lady drove over to Redhill, and while she paid a call there, and left her carriage standing in the town, the coachman got down and drank from the public drinking fountain, went home, and in a few days was dead. It could not therefore be anything in the locality, since here was a fatal result to a stranger who was only in the place perhaps half an hour. One must remember that this had really nothing to do with the question of deep well water or not. The water so-called, which had been supplied many years before, and many years since, was amongst the purest of the deep chalk waters, and the evil arose from an accident in one sense which might have befallen the New River. It had nothing to do with the source of supply. The general question had been touched upon in a way which was often done, and a great deal had been said about the iniquity of London water companies. Now, in the autumn of 1883, whilst travelling in the United States, he made it his business in every town to



which he went to inquire about the water supply, and he was assured by all the engineers to whom he spoke, with one exception, the village of Poughkeepsie, that there was not a single instance of the water being filtered before it was supplied to the towns. When he asked how that was, he told him that they all had filters in their own houses. Then he asked about the poor people, and those who lived in tenements, and those who had not filters, and he replied that they had not thought about them. He told him that if ever there was an epidemic of cholera there the loss of life would be something frightful. In New York the consumption of water was 80 American gallons (equal to 64 English gallons) per head per diem, but the population had so grown, and the mains were so overtaxed, that the terminal pressure was next to nothing, and you saw in the shop windows hydraulic engines to be put into the basements of houses, to be worked from the feeble pressure from the mains in order to pump up the balance of the water to the top of the building. That was how it was dealt with there, because they dare not enforce proper fittings on the customers, because the popular voice did not like it. In Boston the water was so abominable that every person of respectability purchased it from a private company who brought it round in casks. To come back to London, there was an unimpeachable source of supply in the Kent Waterworks, and he thought also an unimpeachable source in the New River. They had heard tales about the intake of the New River which proved to be untrue, and with regard to the supply from the Thames, chemistry had failed to indicate any source of disease. Dr. Frankland said so far as he knew no harm arose from drinking healthy sewage, but, of course, if any outbreak of disease arose it might be that evil would follow. Were they to buy up the present companies? In that case he supposed a miracle would happen, as had constantly happened before. You might go into a committee room in the House of Commons and find a number of gentlemen there, including the town clerk and mayor of the town, declaring that of all the vile supplies of water in creation the one supplied by the company was the worst; then they brought forward the joking witness, the scientific witness, the oldest inhabitant, and so on, and at last they made such a dreadful case that the committee suggested to the company, had you not better agree with the corporation. Then they went out into the lobby and came back and said it was settled. The corporation acquired the undertaking, and without any change of any kind, except the mere fact that the company was purchased by the corporation, that dreadful water, which was not fit to be drunk, or even to be looked at, became proper water for the town to drink for ever afterwards. That was a miracle of very frequent occurrence, and he supposed the same thing would happen in London. It might be said that he was only airing the fallacies of Mr. Easton and Mr. Bazalgette, but that fallacy was this: There were used for all purposes, in London, 32 gallons per head per diem, including waste of all sorts, a great deal of which arose from bad fittings, and they were allowed to exist because of the outcry which would arise if they attempted to enforce the use of good fittings. By no possibility could the people use more than two gallons per head per diem for potable and culinary purposes. The experiment had been carefully made, and therefore with four million persons in London the necessary supply for those purposes would be eight million gallons per day. People at once said, What, would you have a dual service, with all the terrible expense and complication? In other towns it was done, in Paris there was a triple service. To

his mind the mistake was in not keeping water for potable and culinary purposes separate. The water for watering the roads and so on did not require to be of such absolute purity, and he thought it would be extremely well at the cost of some five millions to appropriate the chalk water supplies to cooking and drinking purposes by a separate system of mains, and to use the other water, which was quite good enough, for all other purposes. The question would then become very simple. There was ample water in the chalk for drinking purposes for a London of double the size, but he did not think there was ample water in the chalk within a reasonable distance for the supply of the whole metropolis under the present system. Then he would suggest that this dual supply should be made use of for the purpose of extinguishing fires, and this was by no means so absurd as some people seemed to imagine. The whole of the water used in London for extinguishing fires amounted, according to his recollection, to about a day's supply. It was said that it would be using the most expensive water for extinguishing fires, but that would not matter. They could afford to pump eight million gallons of water a day against a head of 400ft., and with that head and a proper system of pipage they could contribute the greatest quantity of water ever wanted for the greatest fire ever known, taking Captain Shaw as an authority.

A MEMBER asked if Mr. Johnson claimed that the filter press would take out germs from the water, and if so how he arrived at that conclusion, because he had already stated that he did not know what the germs were.

Mr. JOHNSON, in reply, said he had stated in the paper that the object was not to take the germs out, and no one could say that it did. The principal thing was to remove organic matter, because it was found, whether the disease proceeded from germs or merely from some specific poison, it was always associated with organic matter. That was the reason why animal charcoal was introduced into the filtering paper. Further, the removal of phosphates was an important matter, as it deprived the lower organisms of the food necessary for their existence, and therefore there was not the opportunity of the accumulation of a mass of decomposing organic matter in the filter itself. With reference to the fact stated by the President, that London was very healthy, he would simply remark that it might be healthy as compared with other towns, but still it was not so healthy as it would be if perfectly pure water were supplied, because there was a great deal of disease resulting from the drinking of bad water, as could be testified by any medical practitioner.

## NOTES ON KIESELGUHR AND ITS TECHNICAL APPLICATIONS.

BY A. HAACKE.

KIESELGUHR, or infusorial earth, is a white, gray, or greenish powder of very low specific gravity, consisting chiefly of the minute silicious shields of diatomaceæ, the beautiful and varied forms of which will be known to everyone familiar with the use of the microscope. Large deposits of fossil diatoms have been traced in many parts of the world, and several mines producing kieselguhr are worked in Europe, but the largest, and those yielding kieselguhr of the purest quality and lowest specific gravity are situated near Naterleuss station, on the railway from Hamburg to Hanover. The kieselguhr is found there from the surface down to a depth of about 150ft., being covered only by thin beds of diluvial and alluvial origin. The upper stratum of this large deposit supplies the white kieselguhr

This quality contains very little organic matter, but some sand; therefore, after being washed it gives a very pure and porous product. The second stratum produces gray kieselguhr, containing very little sand, but sufficient organic matter for calcining it, and the product then is kieselguhr of perhaps the finest quality existing. If the exertions of colour manufacturers to make a colour which will really be imperishable under the influence of strong acids are to be crowned with success, this perhaps is the material which might lead to satisfactory results. The lowest and by far the largest stratum, varying from 50 to 100 feet in thickness, supplies the green kieselguhr, which contains up to 30 per cent. of organic matter, showing clear imprints and fragments of fishes, well-preserved fir cones, leaves, bark, and twigs of birches, firs, etc. In order to utilise the immense deposits of green kieselguhr, kilns have been erected for burning or calcining same. When dry, this material glows like turf or peat, and this is utilised in the calcining process. The kilns,

important property of kieselguhr, which it owes entirely to its great porosity, is its high non-conducting power. All porous materials are more or less non-conductors, and those are the best which contain in a given square the greatest number of minute cells, enclosing air, which is prevented from circulating. The cooling of heated bodies may be effected by three methods—by radiation, by contact of cold air, and by conduction. A closed air space around a vessel containing high heat is already a good protection, but if the space, instead of with air, were filled with powdered kieselguhr, say 2 in. thick, the insulation would be almost perfect, for the radiation would be totally prevented. The air contained in the kieselguhr would be warmed in a few minutes, but being unable to circulate it would be prevented from giving its heat off. Conduction would be impossible, as the following test will show. If a piece of kieselguhr 2 in. long, by means of a blowpipe, is heated to white heat on the one end, no increase of heat will be

Date.	Temperature of external air.	Pressure.	Steam condensed in 6 hours.		Difference.	Weather.	
			Bare pipe.	Covered pipe.			
October 29.....	Fah. 58.1	lb. 58.3	lb. 270.1	lb. 75.4	195	E. winds. Foggy.	
November {	28.....	55.3	58.9	275.9	76.4	199.5	S. and S.W. winds.
	13.....	43.8	57.3	278.1	79.4	198.7	Strong S.W. wind.
October 23.....	57.1	56	339.2	88.5	250.7	W. wind. Slight rain.	
November {	5.....	49.2	55.1	359.4	92.5	266.9	W. and N.W. winds.
	20.....	48.4	52.8	363.4	97.5	265.9	Moderate W. and S.W. winds.
October 18....	54.5	58.3	441	103.5	337.5	Very strong S.W. winds.	
			2337.4	613.2	1714.2		

simple round furnaces, about 15 feet high by 6 feet diameter, are filled and lighted at the bottom, no additional fuel being required to keep them going. They are continually replenished with green kieselguhr at the top, and the calcined taken out from the grates underneath. The product is perfectly free from moisture and organic matter, and has therefore a much higher market value than green kieselguhr. Its reddish colour is due to some traces of oxide of iron. Kieselguhr has many valuable properties, which adapt it admirably for various industries. It consists almost exclusively of silica, and is therefore in its pure state as fireproof as any material in the world. It resists the action of the strongest acids, but it can be easily made to melt after being mixed with an alkali or some sort of alumina. Even by boiling under pressure combination may be effected. Silicate of sodium or water glass has been made of it in this way, but white sand is now generally used for this article, because kieselguhr has risen considerably in price, and owing to its low specific gravity very large melting pots had to be employed. Its great porosity, although a drawback for this particular industry, makes it very valuable for the manufacture of dynamite. German manufacturers have at times produced dynamite of 82 per cent.—namely, 18 lb. of kieselguhr were able to absorb 82 lb. of nitro-glycerine, and keep it in a firm paste, which did not allow any nitro-glycerine to escape. In England only 75 per cent., i.e., dynamite, is allowed to be made or sold; therefore the green kieselguhr, after being calorised, is best adapted and generally used for this purpose. A very

noticed on the other end. Kieselguhr is now being used by safe makers, cooking stove and kitchener manufacturers, to make fireproof rooms and doors, for filling up bulkheads in steamers, etc. Glass manufacturers use it for annealing, and no doubt the more it becomes known the more will opportunities be found for adopting its use. Its high non-conducting qualities, together with its property of melting when mixed with a slight percentage of alkali, has induced Dr. Frank, of Charlottenburg, in conjunction with Mr. Berkefeld, to take out patents for the manufacture of fireproof refractory bricks, slabs, etc., of very low specific gravity. The present specimen is not quite perfect yet, as the experiments have not been brought to a close. There seems, however, but little doubt that these bricks will be of great importance for the setting of steam boilers, the lining of blast furnaces and the hot-air pipes connected with them, for the backs of fireplaces, and for all purposes in general where it is necessary to prevent the partial escape of heat before being utilised. Small rods are formed of the same material. These are used for absorbing a new disinfectant, namely, bromine, which thus solidified takes 72 times as long to evaporate as in its natural state as a liquid. The small rods saturated with bromine are supplied and kept in glass bottles, which have only to be opened to allow the bromine vapours to escape. I am not aware whether this article is known already in England in this form, but shall be glad to give further information to anyone interested in it. The non-conducting capacity of kieselguhr is



without doubt the highest in its natural state of a powder, but as in this form its application to steam-heated surfaces presents certain drawbacks and difficulties, a composition has been made of it, for about five years past, which can be applied direct to steam-heated surfaces, and is known under the name of kieselguhr, or fossil-meal composition. The amount of heat lost from steam-heated surfaces is hardly fully estimated. Some tests made by the National Smoke Abatement Institution lately, and published in the *Engineer* of January 25th, 1884, fols. 65-66, give valuable information regarding this. A 5in. steam-pipe, 36ft. long, filled with steam of about 55lb. pressure, condensed in 42 hours 2,327·4lb. of water; and another one of same dimensions, but covered  $\frac{1}{8}$ in. thick with some fossil-meal composition, gave 613·2lb. of water, showing a saving of 1,714·2lb., or if 1lb. of steam coal had been used to evaporate 8lb. of water into steam

$$\frac{1714 \cdot 2}{8} = 214 \cdot 2 \text{ lb.}$$

of steam coal were saved in 42 hours, or in the year, if the pipe were hot day and night,

$$214 \cdot 2 \text{ lb.} \times 208 = 44,553 \cdot 6 \text{ lb.}$$

or nearly 20 tons of steam coal were saved. The surface of the bare pipe measured 615 square feet. There are many more industries in which kieselguhr might be used with advantage now that large and regular supplies are attainable. It is used by manufacturers of ultramarine, by soapmakers; a composition for preventing rot and fungus in buildings is made of it; it is a splendid polish for metals, etc.; it could be used for making artificial meerscham, porous pottery, an equivalent for animal charcoal, etc.; and if chemists and manufacturers would experiment more with it, very likely many other uses for it would be discovered.

I have been reminded since I have come into the room that the absorptive property of kieselguhr has been utilised by Messrs. Vorster and Grüneberg in a patent taken out in Germany. What is termed "dry sulphuric acid" is prepared by saturating dry and finely-divided kieselguhr with three or four times its weight of 66° sulphuric acid. The product, which therefore represents at least 75 per cent. of its weight of absolute acid, is said to retain the pulverulent form, and the advantage claimed is that in this condition it can be transported by land or sea in lead-coated sheet iron vessels without damage of breakage. This being so, there are many obvious applications of the saturated kieselguhr.

UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL,  
March 5th, 1884.

MR. E. K. MUSPRATT IN THE CHAIR.

## THE LOSS OF NITRE IN THE MANUFACTURE OF VITRIOL.

BY GEORGE ESCHELLMANN, PH.D.

It has always been an important question to the practical and theoretical chemist to account for the losses of nitre in the manufacture of vitriol. To the former on account of the great value of it, to the latter on account of the interesting reactions taking place between sulphurous acid, nitrous gases, oxygen, and steam. Theoretically the same quantity of nitre should convert an infinite quantity of sulphurous acid into vitriol, provided no losses take place; and in order to reduce them as much as possible various improvements have been introduced in this manufacture. To trace out these losses in the various stages of the process is the object of this paper, and all experiments mentioned have been made on the vitriol chambers in our works (James Muspratt and Sons) in Widnes, and Mr. E. K. Muspratt has kindly allowed me to use such figures as would be useful to my purpose. The losses of nitre are of two kinds first, mechanical losses, such as nitrous gases kept in solution by vitriol used for salt-cake making, and

### NO. 1 SYSTEM.

	Chamber Space— cubic metre per kilo. of S.	Nitre used— per cent. on S.	Oxygen at Outlet, per cent.	Unabsorbed of nitre used per cent.
Quarter I. ..	1·34	3·21	6	10·7
Quarter II. ..	1·47	2·75	7	11·5

### NO. 2 SYSTEM.

	Chamber Space— cubic metre per kilo. of S.	Nitre used— per cent. on S.	Oxygen at Outlet, per cent.	Unabsorbed of nitre used per cent.
Quarter I. ..	4·34	3·24	6·1	12·02
Quarter II. ..	1·47	2·75	7	12·5

nitrous gases escaping unabsorbed at the Gay-Lussac tower and through leakage of the chambers, which latter, if any, should not exceed 2 per cent. of the total loss; secondly, chemical losses through reduction of nitrous gases to nitrogen and nitrous oxide, which compounds are lost to the process. The mechanical loss at the Glover tower is in the vitriol for salt-cake making. An average sample taken from it was 130° Tw., and analysed took 3c.c. of  $\frac{1}{10}$  normal permanganate solution per 10c.c. of vitriol; but permanganate oxidising sulphurous acid as well as nitrous anhydride, the same sample was tested in the nitrometer, where it showed no appreciable reaction, consequently the permanganate was used for oxidising sulphurous acid and arsenious acid, and no nitrous anhydride was present. The mechanical loss by non-absorption of soluble nitrous gases at the Gay-Lussac tower was found out by means of a continuous test of the escaping gas. The gas was drawn by a Bunsen pump through the absorption bottles, and measured by a gas meter. To absorb the gas, it was drawn through a measured quantity of pure vitriol of 1·84 specific gravity, which absorbs all soluble compounds of nitrous gases, and part of this was tested in the nitrometer. Simultaneously a second continuous test was taken by means of an aspirator, all soluble compounds absorbed by caustic solution, and the remaining gas was tested for oxygen. Thus knowing the amount of oxygen and the quantity of pyrites burnt, it is an easy thing to calculate the amount of gas

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S. M. Harrison.	G. T. J. Wells.

Hon. Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be made to Edward George Ballard, Queen's Park, St. Helens.

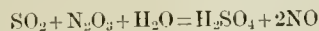
The next meeting of this Section will be held on Wednesday evening, 2nd April, 1884, at Seven o'clock, when the following papers will be read:—1. "Methods of Sampling Chemical Substances," by Mr. A. Norman Tate. 2. "The Damage Done to Vegetation by some Industry Gases," by Dr. Hamburger.

escaping in 24 hours from the system. In our works we have two Gay-Lussac towers, each one connected with a system of chambers burning about the same quantity of pyrites. This test is taken regularly every day, and therefore I am able to give average results for several quarters, as shown in accompanying table. Besides the loss of soluble nitrous gases a certain amount of nitric oxide escapes at the Gay-Lussac tower. To test it the same continuous test as applied to the estimation of the soluble gases was used. The gas was first drawn through strong pure vitriol, of 1.84 specific gravity, to absorb all soluble nitrous gases, then passed through two bottles filled with caustic solution to absorb any sulphurous gas, and then drawn through four bottles filled with permanganate solution and great excess of sulphuric acid. I found it necessary to divide the permanganate solution into four bottles to oxidise all nitric oxide in the escaping gas, on account of the small quantity in it. The nitric oxide is oxidised to nitric acid, and after a duration of 24 hours for each test the contents of the four bottles were collected, the excess of permanganate reduced by a proto-iron solution, and a measured quantity of it boiled with a weighed quantity of double-iron salt. The excess of double-iron salt was titrated back with permanganate solution, and then the quantity of nitric oxide per number of cubic feet was obtained by calculation. This loss in a great number of tests came to 7 per cent. with the chamber space of 1.47 and 7 per cent. of oxygen. The variation of all tests was within 6 and 8 per cent. Therefore the mechanical losses amount to 22 per cent: (1) At Glover tower, trace below 1 per cent. (2) At Gay-Lussac tower, 19 per cent.; (a) through non-absorption of soluble gas, 12 per cent.; (b) through escape of nitric oxide, 7 per cent. (3) Through leakage, etc., 2 per cent. It will be necessary to add that the amount of sulphur going away at Gay-Lussac tower was below  $\frac{1}{2}$  per cent. of the sulphur charged at the kilns. The two Gay-Lussac towers were 1.6 per cent. of the total chamber space, and the strength of the nitrous liquors was about 1 per cent.

I think it will be here the best opportunity to mention what Prof. Lunge and Dr. Naef have found out about chamber escapes in the vitriol process, published in the German *Chemical Industry*, January, 1884. Dr. Naef worked the chambers very yellow, and testing the escapes then found no nitric oxide, no sulphurous acid, but a large quantity of soluble nitrous gases, amounting to 50 per cent. of the total loss; and also there was a certain quantity of nitrogen dioxide present in the gases entering the Gay-Lussac tower. Keeping now the chambers too light—that is to say, a relatively large quantity of sulphurous acid entering the tower—the escape of soluble compounds was reduced to traces, and 22 per cent. of the loss as nitric oxide found in the escapes. Although the first figure seems to me very high it shows evidently that working chambers with either extreme nitrous or sulphurous acid nitre is lost considerably in both cases. In the first case it is lost as nitrous anhydride, in the second as nitric oxide. In ordinary working chambers with a slight escape of sulphurous acid there is no nitrogen dioxide present, but always, as shown previously, a certain amount of nitric oxide (7 per cent.), which is lost. Consequently the best way to avoid losing much nitre at the Gay-Lussac tower is to have all nitrous gases entering it as nitrous anhydride—no mono- or di-oxide being present at all. This can be obtained in keeping the chambers very rich in nitre, allowing no appreciable amount of sulphurous acid to enter the tower, and thus having only nitrous anhydride and nitrogen dioxide; the latter must be reduced by adding sulphurous acid

sufficient to reduce it to nitrous anhydride. If the correct amount of sulphurous acid could be insured this process promises to save nitre. Besides this process, known as Messrs. Benker and Lasne's, the increase of chamber space will give the same result as regards the gases entering the Gay-Lussac tower. It converts all sulphurous acids into vitriol without great excess of nitre and gives the nitric oxide time for oxidation.

So far the losses of nitre accounted for come to 22 per cent., using 2.75 per cent. on the sulphur burnt, and with 1.47 cubic metre of chamber space per kilo. of sulphur. The chemical losses can only take place through reduction of nitrous gases to either nitrogen or nitrous oxide, which gases are insoluble in strong vitriol, and lost to the process. As we have no proper reliable test for nitrous oxide, and estimation of nitrogen produced by reduction being out of question, I was only able to show by experiments made on vitriol chambers under which conditions these reductions are likely to take place. The question of reduction in the Glover tower has been the object of several papers, and theory and practice have come to the conclusion that there is not much, if any. In the Glover tower we have nitrous vitriol or nitric acid going down and meeting hot sulphurous acid and air. No reduction takes place below 200° C., according to Professor Lunge's paper published in *Dingler's Polytechnic Journal*, vol. 225, 1877. The highest temperature of the vitriol running off the tower which I have observed was 150° C. The only possibility is just at the inlet of the gases from the kilns, but when the vitriol reaches that part it is already denitrated. The upper part denitrates, the lower part boils down, and the reaction in the tower is represented by—



According to Professor Thomsen's book, "Thermochemical Investigations," the heat produced for  $\text{H}_2\text{SO}_4$  by sulphurous acid, oxygen, and water, is 53,480 calories. In the first chamber drips are generally kept about 130° Tw. This corresponds with the formula  $\text{H}_2\text{SO}_4 + 2$  parts water. According to the same authority 9,418 calories are evolved in combining  $\text{H}_2\text{SO}_4$  with 2 parts water, therefore this produces a total quantity of 62,898 calories. For 134 grms. sulphuric acid of this strength 54 grms. of water must be introduced, and this is done generally as steam of 125° C. 1 grm. steam of 125° C. contains 644.625 calories—thus 54 grms. = 34,809 calories. Assuming the quantity of steam to be increased by  $\frac{1}{2}$ —that is, producing  $\text{H}_2\text{SO}_4 + 3$  parts water—then 18 grms. of steam of 125° would be required in addition. 18 grms. steam contain 11,603 calories; to combine ( $\text{H}_2\text{SO}_4 + 2$  parts water) with 1 part water = 1,729 calories are evolved. The temperature of the first chamber will be of an average of 50° C., therefore the steam will be cooled to 50° C. 1 grm. water of 50° contains = 50.087 calories; 54 grms. contain = 2,704 calories; 18 grms. contain = 901 calories. Thus for 54 grms. 34,809 - 2,704 calories = 32,105 calories, would be evolved; and for 18 grms. 11,603 - 901 = 10,702 calories, would be evolved. Further, for  $\text{H}_2\text{SO}_4 + 2$  parts water would be therefore evolved, for reaction = 62,898; from 3 parts water as steam = 32,105; total, 95,003. For  $\text{H}_2\text{SO}_4 + 3$  parts water = 95,003 + 10,702 + 1,729 = 107,434, would be evolved. Then having for 100 volumes per cent. of gas entering the chamber containing 6 volumes per cent.  $\text{SO}_2$  and 94 per cent. of other gases, in the first case 3 x water = 18 volumes per cent., must be introduced; in the second case 4 x water = 24 volumes per cent. Thus we have for 118 volumes evolution of 95,003 calories; for 124, evolution of 107,434 calories; or for equal volumes (1) 95,003 : 118 = 805 calories; or for equal volumes (2) 107,434 : 124 =



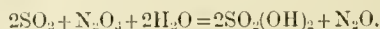
867 calories. The temperature excess of the first chamber over the air is proportional to the quantity of heat evolved, the temperature of the chamber being 50° C., and producing vitriol with three equivalents of water evolve 805 calories per unit of volume, and have an excess of temperature over air of 50 - 15 = 35°. Increasing the steam by one equivalent, and producing 876 calories per unit, we have the following proportion to ascertain the increase of temperature: 805 : 867 = 35° :  $x$ .  $x$  = 37·6°. Consequently an increase of steam by one-third would raise the temperature in the first chamber by 2·6°C., but such variation never happens in well-working chambers. I come therefore to the conclusion that the variation of temperature caused by variation of steam can be entirely neglected, being practically none. The variation of the temperature caused by the surrounding air I found to be within 5°C. in 24 hours, increasing towards the warmer part of the day; and this variation will be rather lower in winter, when the variation of the outside temperature is generally less than in summer.

The only source for variation of temperature is the formation of vitriol, and therefore we have in the observation of the temperature always a quick indicator of the quantity of vitriol made in the first chamber. This quantity depends to the greatest part on the quantity of nitrous gases present, and I have proved this by experiments made on our chambers and by observation of their temperatures. In our works we have two systems of chambers burning about equal quantities of pyrites. The first system consists of one set of five chambers with one Gay-Lussac tower, the second system of two sets of three chambers and two chambers respectively, both joining into a second Gay-Lussac tower. The nitrous liquor obtained from the Gay-Lussac tower is sufficient to supply the two small sets completely with nitre, and all necessary potting is done at the large system, which receives a very small quantity of nitrous liquor. Consequently the two small sets receive a continuous supply of nitrous gases and the large one a periodical one, although the potting is divided over as many intervals as possible. The two small sets show no variation of temperature except what is due to the variation of the surrounding air; the large set shows a very interesting variation, which is entirely due to the potting. This is done every two hours, and each time before potting, when the nitrous gases are lowest in amount in the first chamber, the temperature shows its lowest, rising slowly with the nitrous gas coming of the fresh pots. This variation extends from 48° to 68° C., and repeats itself regularly, showing evidently that the temperature depends on the amount of nitre present, and as the temperature depends on the quantity of vitriol made, a fall of temperature indicates less vitriol made, owing to scarcity of nitre, provided the amount of oxygen is right, which will be shown by the test of the escaping gases after the Gay-Lussac tower. The thermometers were inserted 22ft. distant from the inlet of the gases at each set, and the gas entering it was about 35° C. in autumn and winter. The temperature of the small sets was from 50° to 53° C.

To prove further the influence of the quantity of nitrous gases present, I missed in an experiment the potting of nitre once at the large set, leaving the small quantity of nitrous liquor at the Glover tower untouched as well as the steam. The temperature fell to 41° C. The temperature of the entering gas was 35° in this case. The difference of 6° between 35° and 41°, is owing to the small quantity of vitriol made by the small quantity of nitrous liquor running down the Glover tower. The drips were—

	I.	II.	III.	IV.
Before starting.....	131	129	116	108
Lowest during experiment....	114	112	101	91

and large escape of sulphurous acid was observed at the Gay-Lussac tower. The chambers worked very irregularly after the experiment, until not only the missed nitre was put in, but an additional quantity of it, to compensate for reduction of the nitre, through excess of sulphurous acid on the small quantity of nitre present.



The quantity of nitre missed during experiment was 80 per cent. of the whole quantity entering per two hours. Another experiment with too much nitre was made at the small set, where no variation of temperature takes place. The temperature rose 9°, and the strength of vitriol increased from 134° to 142°, showing more vitriol was made. However, it is not advisable to work with a great excess of nitrous gas, on account of the losses increasing then by bad absorption at the Gay-Lussac tower, as shown previously. These experiments with nitre show the bad effect caused by shortness of it; and judging from the regular variation of temperature at the large system, it is certainly of advantage to have a continuous supply of nitrous gas entering the chamber to avoid reduction, which must take place to some extent when the nitrous gas is lowest in amount just before potting time. This is the reason the Continental works prefer using nitric acid.

Professor Lunge published experiments on the influence of steam on nitrous gas in *Dingler's Polytechnic Journal*, vol. 243. Mixing sulphurous acid, air, water, and nitric oxide above mercury there was always very considerable reduction to nitrous oxide, not to nitrogen. Mixing these gases above sulphuric acid of 1·455 specific gravity of 14° C., no reduction took place. This shows considerable reduction takes place with excess of water and no sulphuric acid present; and this condition is fulfilled in the chambers at the inlet of the steam. In the first chamber generally acid of 134° is produced—that is, sulphuric anhydride with three equivalents of water. It is rather a hopeless task to find out by experiments on working chambers how much nitre is reduced by the three equivalents, because vitriol above 134° begins rapidly to destroy the lead of the chamber and to dissolve the nitrous gases. However, to find it out, I worked one set of chambers with four equivalents of water—that is, producing vitriol of about 114° instead of 134°. I chose for experiment one of the small sets, which receives a continuous supply of nitrous gas by nitrous vitriol. On account of this the temperature is very steady, and any change of it could be immediately observed. This set consists of three chambers, and during the experiment the vitriol produced in the two last chambers was kept the usual strength, everything else left the same as well. Each egg of nitrous liquor running down the Glover tower was tested for nitre to ascertain any variation of strength, which was of no consequence. The nitrous gas dissolved in the vitriol was equal to 1,852lb. of nitre in 96 hours, representing 8·7 per cent. of nitre on the quantity of sulphur charged (48 per cent. of pyrites). I have shown in the previous calculation, that an increase of one-third of steam, which is equal to the difference between 114° and 134°, could only increase the temperature by 2·6°, provided the same quantity of vitriol was made. The temperature before starting was 50°, and it rose at once to 53°, to remain constant for a few hours, when it began to fall slowly to 46°, which fall took 48 hours. Nitre had to be introduced, which rose the temperature to 54°; the same slow fall of temperature repeated itself within the next 48 hours, when again nitre had to be potted. After 96 hours' duration the steam was put to its former standard,

and the extra nitre used weighed 60lb. For this set 1,852lb. were used during the 96 hours, consequently the 60lb. represent a reduction of 3·2 per cent. of this quantity, and 10½ per cent. of the lost nitre, using 2·75 per cent. The set worked very regularly during the experiment, and the reduction which took place was very gradual. According to this, 10½ per cent. of the nitre was reduced, producing vitriol with four equivalents of water in the first chamber. As reduction only takes place at inlets, as proved by the already-mentioned experiments of Professor Lunge, in *Dingler's Polyt. Journal*, the same amount must be reduced for the first three equivalents, being therefore 31·5 per cent. of the lost nitre.

There has always been much difference of opinion as to with what amount of oxygen the chambers ought to be worked. Dr. Hurter finds in his paper, published in the *Journal of the Society of Chemical Industry*, 1883, 6 to 10 per cent., according to working conditions. It depends, in my opinion, greatly on the available chamber space. I had opportunity to work chambers with different chamber space, and I found the results shown in this table could always be obtained, and remain within 0·5 per cent. loss of sulphur at the Gay-Lussac tower.

Chamber space.		Oxygen. Percent.		Nitre per cent. on Sulphur.	
1'	....	4½ to 5	....	4'	
1½	....	5	5½	....	3·7
1½	....	5½	6	....	3·5
1½	....	6	6½	....	3·1
1½	....	6½	7	....	2·8

If I tried to work the chambers much higher in oxygen, as represented in this table, sulphurous acid was carried away mechanically at the Gay-Lussac tower, and increased the loss of sulphur considerably, sometimes to about 2 or 3 per cent. Great excess of oxygen shows its effect at once at the temperature of the first chamber, which when it reaches over 10 volumes per cent. goes down to nothing much above the temperature of entering gases.

If the oxygen goes below 3 per cent. a great quantity of extra nitre must be used, which amounts in my experience up to 6 or 7 per cent. on the sulphur burnt. In this case a great amount of nitric oxide escapes at the Gay-Lussac tower, which then does not require any testing, but will show itself by red fumes at the outlet of the chimney, caused by the oxidation of the monoxide to nitrous anhydride, which reaction ought to have taken place in the chambers. This will explain the disadvantage of a small chamber space. In a small chamber space most of the vitriol must be made in the first part of it. In order to do this the circulating nitre per quantity of sulphur must be increased, likewise the chambers must be worked with less speed, which means less oxygen. Then the disadvantage of working with more circulating nitre is increased through shortness of oxygen, the greater quantity of nitric oxide is mixed with a smaller quantity of oxygen, therefore not oxidised so readily, and particles of it are more likely to be reduced by sulphurous acid to nitrous oxide. Besides this there is much less time given for its oxidation; proof of this that even with 1·47 of chamber space, still 7 per cent. of nitric oxide escaped unoxidised from the Gay-Lussac tower in the presence of 7 per cent. of oxygen. Working with large chamber space less vitriol is required to be made in the first chamber, the formation of it being divided over a larger space; the chambers can be worked with less circulating nitre and with more oxygen; and more oxygen being present for a smaller quantity of nitre, the monoxide is more readily oxidised and likewise allowed more time for

its oxidation. In our works the 2·75 per cent. of nitre used divides itself as follows—

Gay-Lussac tower	.....	0·330 or 12	as soluble nitrous gases
Gay-Lussac tower	.....	0·192 or 7	as nitric oxide
Glover tower, leakage, etc.	...	0·082 or 3	
		0·866 or 31½	from steam at inlets
		1·470 or 53½	accounted for

This leaves 46 per cent unaccounted for, which is due to the insufficient chamber space, to the irregularity of introducing the nitre, and to inevitable accidents, which are all included in the 2·75 per cent.

Before concluding this paper I should like to say which will be the lowest quantity of nitre by which vitriol could possibly be made. To this purpose would be required a chamber space of about 1·8 or 2 cubic metres per kilo. of sulphur, a regular stream of nitrous gases entering the chambers, strength of vitriol and oxygen kept right, and a sufficiently large Gay-Lussac tower, to allow no more than 0·33 per cent. of the soluble nitrous gases to escape. The losses would then divide themselves as follows:—

Gay-Lussac tower	.....	0·330 = 26
Glover tower, etc.	.....	0·082 = 6
Steam at Inlets	.....	0·866 = 68
		1·278 = 100

making no allowance for inevitable irregularities.

#### DISCUSSION.

Dr. HURTER said the paper was an exceedingly interesting one. Many years ago another of Messrs. Muspratt's chemists, Mr. Vorster, made investigations into the losses of nitrate occurring in the manufacture of vitriol, and the results arrived at by Mr. Vorster bore out Dr. Eschellmann's remarks as to the very large amount of nitrate put into the system which could not be accounted for. Mr. Vorster came to the conclusion that the main loss occurred in the Glover tower. Shortly after Mr. Vorster's paper appeared the Noxious Vapours Manufacturers Committee commenced its operations, and he (Dr. Hurter) made a number of experiments very similar to Mr. Vorster's, from which he arrived at a similar conclusion, viz., that the denitration of nitrous vitriol could not be carried out without loss. On the other hand, Professor Lunge now maintained that the whole of the nitrate was probably lost in the Gay-Lussac tower. Professor Lunge, in the paper to which Dr. Eschellmann had alluded to, said that with a proper construction of chambers and skilled working, but only under these conditions, the chemical losses unaccounted for were, if not absolutely zero, far too small to be taken into consideration; but this would not apply in works where more than 3 per cent. of nitrate was used. So that Professor Lunge's position now was, that where only 3 per cent. of nitrate was used the greater proportion of loss could be accounted for. Dr. Eschellmann had told them that all the loss accounted for by Dr. Lunge amounted to 50 per cent. of the total nitrate used. Looking at Professor Lunge's experiments more carefully, he (Dr. Hurter) found them to be such as could not be accepted as normal experiments. The amount of nitrate used must certainly have been more than that on which the loss had been calculated; and he objected to the experiments because the amount of nitrogen compounds escaping at the Gay-Lussac were abnormally large. Dr. Eschellmann's observations agreed entirely with what Mr. McTear, Mr. Jackson, and himself had published years ago, viz., that there was certainly somewhere in the system a loss of nitrogen compounds which could not be discovered by the methods of analysis at present at disposal. It was asserted in reply to this that their methods of analysis were bad, so that they



could not discover the amount of nitrate leaving at the Gay Lussac. He agreed that they were bad if they only showed 80 per cent. of the quantity passing away, but nevertheless this did not affect the general conclusion that there was a very large amount of nitrogen compounds lost. Dr. Eschellmann had endeavoured to show where the loss occurred; and had stated that the temperature of the chamber was an index to the amount of vitriol made in the chamber. He entirely agreed with Dr. Eschellmann in this, and he believed that the amount of steam introduced and the air outside had comparatively small influence upon the temperature. On the whole Dr. Eschellmann had shown a state of affairs very much the same as he himself and the other chemists he had named had found some time since. It was very important to find out where the loss took place; and supposing  $31\frac{1}{2}$  per cent. to be lost in the chambers, Dr. Eschellmann pointed to the fact that it was perfectly useless to attempt to find methods to work with a small chamber space, which meant working with a large amount of circulating nitrate; but he could not entirely agree with the method by means of which that loss in the chambers was established. The experiment had lasted only a short time, and the amount of nitrate potted was a small fraction of the whole nitrate in circulation, which, to his mind, rendered the conclusion doubtful.

Dr. HAMBURGER: The question of the consumption of nitre in O.V. chambers is as interesting as important to the manufacturer. I have myself made experiments on that subject, and my figures very closely agree with those of Dr. Eschellmann. The so-called mechanical loss, excluding NO, amounts in our works (Messrs. Gamble, St. Helens) to about 22 per cent., chamber-room and nitre consumption being nearly the same as stated by the lecturer. I also believe that the excess of water, or rather steam, in the chambers has a great deal to do with the reduction of N compounds to  $N_2O$ . I think it was R. Weber who first pointed out this as a possible source of loss ( $N_2O_3 + 2SO_2$  and  $2H_2O = N_2O + 2SO_4H_2$ ); but another loss, which might be very considerable, is the escape of NO and  $N_2O$ . I have failed in determining NO with permanganate, as suggested by Prof. Lunge. Samples drawn at the same time from two openings of the chamber escape tunnel, close to each other, gave different and therefore unreliable results. Another cause of loss was suggested by Mr. Davis, some years ago, as taking place in the Gay-Lussac tower by the oxidation of  $As_2O_3$  into  $As_2O_5$ . It was rightly observed at the time that this cannot be serious, as works making sulphuric acid from brimstone, and working with Glover and Gay-Lussac towers, do not experience this loss; they should, consequently, show a great reduction of their nitre consumption, which is not borne out in practice. I am not satisfied with the explanation given by certain investigators, who think they can account for nearly all the nitre lost in the manufacture of O.V., but am rather of opinion that there is something going on within the chambers of which we at present know nothing, and which causes the reduction of nitrogen compounds into such which we cannot recover. In conclusion, I wish to say that the principal points for a low nitre consumption are increased chamber space, a small amount of circulating nitre combined with sufficient absorption plant, and, as Dr. Eschellmann showed, regularity in the supply of nitre. With regard to "chemical loss," I quite endorse the words of Mr. Mactear (*Chem. News*, 1879, page 232): "The unaccounted-for loss is intimately connected with the working of chambers, as in most cases it is found that where the percentage of nitre used on sulphur is high, so also is the loss of nitre unaccounted for calculated on sulphur."

## REPLY.

Dr. ESCHELLMANN, in replying, thanked the meeting for the kind attention given to his paper. He was much pleased that Dr. Hurter's experiments agreed with his own.

The CHAIRMAN said they were much indebted to Dr. Eschellmann for his paper, which had been a very interesting one, and had cleared up some doubtful points in the sulphuric acid manufacture. The discussion on the question had been going on for some time, having been started by some experiments made by Mr. Vorster at the works with which he (the chairman) was connected. Undoubtedly some of Mr. Vorster's conclusions were wrong. He based his calculations too much upon experiments in the laboratory rather than in the chambers themselves. As to Professor Lunge, it was evident that he had worked with an excess of nitrate. A number of experiments had been made by the Committee of the Alkali Manufacturers' Association at ten works, and from calculations he had made—assuming, which he did not think would be far wrong, that the average amount of nitrate used in those experiments was 4 per cent.—he found the total escape in grains of nitrogen per cubic foot would represent somewhere about 20 per cent. of the total loss, owing to the non-absorption in the Gay-Lussac tower.

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## ON THE ACTION OF NITRATES ON ALKALINE SULPHIDES.

BY E. W. PARNELL.

In the spring of 1882 I read a paper before this Section of our Society, giving an account of some experiments I had made on the action of nitrates on alkaline sulphides at different temperatures in an alkaline solution, the object being more especially to investigate the formation of ammonia, which different authorities had represented to be produced from the nitrate at certain stages of its reduction. From my experiments I ascertained in the first place that the proportion of ammonia evolved during the oxidation of crude Leblanc liquors is very far below what would be produced if the action that takes place had been correctly represented by the authorities above referred to; and secondly, that when especially prepared solutions are employed, in the place of the crude Leblanc liquor, oxidation of the sulphide goes on without any formation of ammonia whatever. From this I assumed that the ammonia evolved from the Leblanc liquors arose from the decomposition of cyanides, but remarked that the subject called for further investigation. Since then the matter has been further investigated by Professor Lunge and Mr. J. H. Smith, an account of an elaborate series of experiments having been published by them in the numbers of our Journal for November and December last. In these experiments the authors follow closely the action of the nitrate through its various stages, and investigate in an exhaustive way the gradual oxidation of the sulphur compounds under greatly-varying conditions. The conclusions at which they arrive with regard to the formation of ammonia, and to which I wish now especially to draw attention, are, first, that when the action takes place on the large scale in the manufacture of caustic soda by the Leblanc process, ammonia is produced from the nitrate by the reducing action of the metallic iron of the vessels in which the operation is conducted; secondly, that a small proportion of nitrate is reduced to ammonia by the action of the sulphide, in absence of iron; and thirdly, that when metallic iron is present the action of the

nitrate on the sulphide is hastened by the action going on between the iron and the nitrate. The figures that the authors give in these two cases are, in absence of iron, of the total nitrate decomposed by the sulphide, 1·4 per cent. is reduced to ammonia; while when metallic iron is present the proportion rises to 10·4 per cent. It will be seen, therefore, that in the former case the authors practically confirm what I had pointed out, namely that the formulæ given by earlier authorities, in which ammonia was represented as being produced to a considerable extent, are incorrect. The conclusions, however, at which they arrive with regard to the part played by the metallic iron cannot in any way be reconciled with some of the results obtained in the experiments I had made. In criticising these results the authors dispose of the differences by simply assuming that my experiments must be "wrong." Now in any case such a conclusion is a very unsatisfactory one. I, knowing the care with which my former experiments had been conducted, might with equal reason assume that the authors' experiments were wrong. I thought it better, however, to investigate further, and, if possible, to discover the cause of the apparent discrepancies, and I now propose to give an account of the results of some experiments I have made with this object. In the first place, the authors speak of the reduction of nitrate by metallic iron in an alkaline solution as if it were a simple matter, and refer to a well-known method of estimating nitrate based on this reaction. Now the only analytical method that I can think of to which they refer is that known as Harcourt and Siewart's, where zinc and iron filings are employed together. In this case, however, even if the iron were able to act alone, any such action would be entirely prevented by the zinc, which at once throws the iron into an electro-negative condition. In this method the use of the iron filings is simply to hasten the solution of the zinc by galvanic action. The authors further say that they have succeeded in reducing as much as 90 per cent. of nitrate to ammonia in an alkaline solution by means of metallic iron. This is to me most extraordinary, as although I have made repeated trials with different proportions of materials and varying strengths of solution, I have in no case been able to get any satisfactory addition of the nitrate. I have found that generally on commencing to boil, ammonia to a slight extent has been produced, but that the action quickly ceases, no more ammonia being evolved until a temperature of 450° to 500° Fahr. is reached, when a further slight reduction takes place. My experiments were as follows: I first boiled a solution containing 50grms. of white fused caustic soda of high strength, and 2grms. of potassium nitrate, with 5grms. of very fine iron wire, in a flask connected with a suitable condensing apparatus. The connecting tube was about  $\frac{5}{16}$  in. internal diameter, and for a length of about 18 in. had a fall towards the flask, so that liquid condensed in it might be returned to the boiling mixture. The object of this was to entirely free the steam from the minute particles of alkali that are always carried away from a boiling caustic solution. On commencing to boil the volume of the solution was about 100c.c. In twenty minutes 42c.c. had been carried over, and the ammonia condensed was only 0·0003grm.  $\text{NH}_3$ . A similar experiment with the same quantities of iron and nitrate gave 0·0006grm.  $\text{NH}_3$ , after twenty-five minutes' boiling. Iron filings give rather better results. 5grms. in a solution of 50grms. of caustic and 2grms. nitrate gave 0·006grm.  $\text{NH}_3$  by the time a temperature of 450° Fahr. had been reached in the decomposing flask, while in another case 0·009grm.  $\text{NH}_3$  by boiling up to 560° Fahr. Even this, however,

only represented 3 per cent. of the nitrate reduced to ammonia. If zinc be added a very different result is obtained, as the following experiment shows: 50grms. of caustic, 2grms. of nitrate, and 5grms. of iron wire, in about 120c.c. of water, were boiled for half an hour, and gave 0·0007grm.  $\text{NH}_3$ . The liquid in the flask was then diluted to its original volume, and 3grms. of zinc were added. Boiling then for thirteen minutes gave 0·1grm.  $\text{NH}_3$ , about 140 times as much as had been produced by the previous half-hour's boiling. Now, on referring to Lunge and Smith's paper, and to their experiments on this subject, at page 461 of the number of the Journal for November last, it will be seen that they employ a caustic solution that yields ammonia on being boiled alone, as before adding iron wire they say they boiled 150c.c. of 90° Tw. liquor for half an hour, and "estimated the ammonia." If they employed crude liquor, sulphide would also be present. How they can therefore, from operations on such a mixture, conclude that their "experiments re-establish the generally well-known fact that iron does reduce nitrate to the form of ammonia in a boiling caustic solution" is more than I can understand. In the first place, the evolution of ammonia from boiling Leblanc liquors does not cease, at all events, at 570° Fahr., as the following experiment shows: 100c.c. of crude Leblanc liquor of 90° Tw. were boiled, and the ammonia collected, with the following results:—

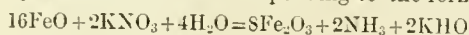
Time. Minutes.	Temperature. F.	Total. $\text{NH}_3$ . Grms.
8	360°	·0066
17	462°	·0102
20	526°	·0137
21	540°	·0165
21½	550°	·0179
22½	570°	·0190

It is quite possible that this evolution may be modified under the changed condition of their experiments. So far, therefore, the difference to which I had previously called attention between the action of nitrate on pure sulphide in a pure alkaline solution, in which case no ammonia is produced, and the corresponding action in Leblanc liquors, cannot be accounted for by the action on the metallic iron. In order to discover the cause of this difference I next proceeded to add to the mixtures, as before prepared, small quantities of the different impurities found in Leblanc liquors. Without going into unnecessary details, the result was that I found that the addition of a very small proportion of iron oxide caused the formation of a large proportion of ammonia—that, in fact, the formation of ammonia is simply due to the action of nitrate on ferrous sulphide, which is oxidised to ferric oxide, and then reduced by the alkaline sulphide to ferrous sulphide again, this action and reaction continuing until one of the reagents is exhausted. In ordinary Leblanc liquors the proportion of iron held in suspension or solution is not sufficiently large to cause much of the nitrate to be reduced to ammonia in this way, but a very small addition of an iron salt increases the proportion enormously. It is strange that this action and reaction should have entirely escaped the notice of Lunge and Smith, as they actually speak of the oxidation of ferrous sulphide by the nitrate, with formation of ammonia, as one of the *probable* stages of the oxidation of the iron. But it seems never to have occurred to them that the ferric oxide would at once be reconverted to ferrous sulphide by the action of the alkaline sulphide. The action on the ferrous sulphide begins at a much lower temperature than the corresponding action on pure alkaline sulphide, although it is very feeble under about 370° Fahr. This action and reaction, however, together with the fact of any oxidation of ferrous sulphide going on at less than 370°



Fahr., is quite sufficient to explain the differences between the results of Lunge and Smith's experiments and those of my former ones; and which differences they expressed themselves unable to account for. In criticising my results they seem entirely to have overlooked the fact that I operated on especially prepared pure solutions, and also that in my former paper I had myself especially drawn attention to the difference between the behaviour of such mixtures and Leblanc liquors on the large scale.

I give here a few experiments on the action of the iron sulphide. A solution of caustic soda of 90° Tw. was prepared from pure caustic, and pure sulphide added equivalent to about 2 parts of Na<sub>2</sub>S to 100 parts Na<sub>2</sub>O; 100c.c. were boiled with addition of nitrate up to 550° Fahr., when all sulphide had disappeared and no ammonia had been produced; 100c.c. were next taken, and 1.5gm. nitrate and 1gm. ferrous sulphate added; ammonia appeared at 280° Fahr., was rapidly evolved at 370° F., and ceased at about 500° F., the proportion corresponding to about 88 per cent. of the nitrate employed. In this experiment the nitrate was insufficient thoroughly to oxidise the sulphide. 100c.c. were taken and boiled with 1.5gm. nitrate and 2grms. ferrous sulphate. The proportion of ammonia was not increased, however, and in no case could more than 90 per cent. of the nitrate be reduced to ammonia. In the experiment where 1gm. of ferrous sulphate was employed, the ammonia due to its oxidation amounts to 0.007gm. NH<sub>3</sub>, while the total NH<sub>3</sub> obtained was 0.22gm., thus showing that the iron had acted and reacted some thirty times. In another experiment I operated with a much smaller proportion of ferrous sulphate—0.05gm. to 2.5grms. of nitrate, the latter being slightly in excess of the sulphide. In this case the ammonia obtained amounted to 0.09gm.; that due to the action on the ferrous sulphide was only 0.0003gm., so that the latter had done its work some three hundred times. Ferrous oxide alone in absence of sulphide reduces nitrate to ammonia in a boiling alkaline solution, and in some cases I have succeeded in obtaining very nearly the full amount corresponding to the formula



2grms. of ferrous sulphate, for instance, boiled with 2gm. nitrate and 50grms. caustic, gave 0.0162gm. NH<sub>3</sub> against 0.0152gm. calculated. I find that zinc oxide acts in a similar way to the iron oxide, but not by any means so rapidly. Caustic soda made from liquors that have been freed from sulphide by means of zinc frequently contain a small proportion of the latter, and in one of my experiments I obtained ammonia in absence of iron, which I subsequently ascertained to be entirely due to this. Caustic soda solutions of 90° Tw. will hold up zinc in presence of sulphide, so that in such a case the presence of the zinc may easily escape one's notice.

The conclusions at which I arrive from these and my former experiments therefore are: (1) That oxidation of alkaline sulphide by nitrate in a boiling alkaline solution does not commence below 370° F.; (2) that oxidation takes place without formation of ammonia; (3) that ferrous sulphide, ferrous oxide, zinc sulphide, and no doubt some other metallic sulphides are oxidised by nitrate, in a boiling alkaline solution, with formation of ammonia, and that consequently when any of these, more especially the iron compounds, are present in a mixture of caustic soda, nitrate, and alkaline sulphide, ammonia to a large extent may be produced on boiling; and (4) that metallic iron has a very feeble reducing action on nitrate, with formation of ammonia in a boiling alkaline solution. Now, on referring to the paper of Lunge and Smith, it will be seen that all

apparent discrepancies can easily be explained. They boil the iron wire in a solution containing sulphide. The latter attacks the iron, in this case no doubt producing ammonia from the simple action itself, but by increasing the proportion of iron sulphide in suspension or solution in the mixture, they enormously increase the amount of ammonia obtained when alkaline sulphide is being oxidised by nitrate. Iron is always present to a certain extent in crude Leblanc liquors, consequently as they only experimented on crude liquors they invariably obtained some ammonia. The discrepancy between one experiment of mine and theirs, to which they draw especial attention at page 461, and to which I have already referred, where I found an addition of iron caused no increase of ammonia, is explained by the liquor containing only a very small proportion of sulphide, 0.12 to 100 of alkali, as shown in the analysis. I have therefore no modification to make in any of the direct conclusions I drew from my former experiments, the object of which was as much to investigate the purely abstract question of the action of the nitrate on the sulphide as the actions that go on in the Leblanc liquors. Of the latter I remarked that "by adding a by no means excessive quantity of nitre to the boiling liquid before it reaches 90° Tw., it is possible greatly to reduce the proportion of sulphide, and that under these circumstances sulphite and not sulphate is principally formed. Further, that it is well known that at certain stages of the boiling an addition of nitre is immediately followed by an unmistakable evolution of ammonia." I said that I was "only able at the time to suggest an explanation of these differences, and that is that some cyanogen compound, possibly the sulpho-cyanide, acts as a carrier of oxygen from the nitrate to the sulphide, the cyanogen at the same time being partially split up." What I have, therefore, to modify in my former paper is the negative conclusion, or rather assumption, that all the ammonia was derived from the cyanide in the case of Leblanc liquors.

## Manchester Section.

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J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

On April 1st a paper will be read by Messrs. C. F. Cross and E. J. Bevan "On the Oxidation of Cellulose, with Special Reference to the Chemistry of Bleaching and Printing."

A conference will take place on "Standard Methods of Analysis, Sampling, and Stating the Results."

THE MEMORIAL HALL, ALBERT SQUARE,  
March 4th, 1884.

MR. IVAN LEVINSTEIN IN THE CHAIR.

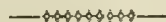
## ON THE DYEING PROPERTIES OF ANTHRACALLOL.

BY DR. R. BOURCART.

I will endeavour to show you this evening illustrations of cloth mordanted with iron and alumina and dyed in anthracallol. This substance has been known for several years, and was discovered by Säuberlich, but it has never been pointed out that it is a

colouring matter. About three years ago Mr. von Georgievics and myself made some experiments on anthragallol, and we prepared several amido compounds of this substance, which formed the subject of a note which Professor Noelting presented to the Société Industrielle de Mulhouse. We did not communicate our discovery about the dyeing properties of anthragallol because at that time alizarin was more expensive than it is now, and there was some hope that it might be useful as a substitute for that colouring matter. Since last year, however, the price of alizarin has fallen very much, owing to the patents which have been taken out for its manufacture having expired, and the difference between the prices of alizarin and anthragallol is accordingly too great at present to permit any hope of the latter being used as a substitute for the former unless somebody finds a way of increasing the yield of manufacture or a means of lowering the cost of substances used in the preparation of this dyestuff. I have the honour to show you two bottles containing anthragallol in a suspended state similar to that in which alizarin is sold. Both bottles contain the same substance prepared by two different methods. The first illustration is the result of the combination between benzoic acid and gallic acid in the presence of sulphuric acid. This combination should rather be called a cementing together of two organic nuclei of benzol. Two molecules of water are eliminated and the well-known double nucleus of anthracene is formed, so we see that anthragallol and alizarin are derived from the same hydrocarbon, which is anthracene. In the case of alizarin, we take anthracene and modify it slightly so as to change it into alizarin, but in the condensation referred to we make anthracene, or at least one of its derivatives, which is anthragallol. The latter could, if required, be easily converted into alizarin or anthracene through chemical reactions so as to prove its construction. My other illustration is prepared in the same manner as the first, only here the substances which combine are phthalic acid and pyrogallol. In both cases, after treating for several hours with an excess of sulphuric acid and letting it cool well, the dyestuff is precipitated by pouring the sulphuric solution into a great quantity of water and washing on a cloth-filter till there is no acid present. Anthragallol contains one atom of oxygen more than alizarin. It is an isomer of purpurin, having the same composition and containing the same percentage of the various elements, carbon, hydrogen, and oxygen as purpurin; but those atoms are placed in a slightly different

gallol gives brown lakes with alumina and black lakes with iron, is perfectly fast to soap, and withstands strong bleaching liquor the same as alizarin colours will do. I have tried to prepare anthragallol in a third manner by starting from nitro-alizarin, and although I have obtained substances presenting great interest from a scientific point of view, I have not been able to transform them into anthragallol. I have now the honour of showing you illustrations of printed cretonnes from Alsace. The large ones have been kindly lent to me by Professor Roscoe. They are from Thierry-Mieg, in Mulhouse. I have also a selection of very fine cretonnes mostly from Weiss-Fries, in Mulhouse. These cretonnes are done partly with machine and partly with block, and some of them first with machine, and then fitted in with block. I draw your attention to those furnitures, two yards wide, done entirely with block. I have also a very fine selection of prints from Switzerland, presented to me by four firms in Glarus. Some of the veils are calendered together four pieces thick, and printed four together with block. They are done with aniline colours, mostly for the Oriental markets. Here are also handkerchiefs, sold in the Oriental markets for head-gear. They are nearly all black, this colour being in great favour amongst the Orientals, and are ornamented round the border with fine aniline colours. Here is also a splendid selection of Swiss turkey-red handkerchiefs, from the works of Colonel Tschudi, in Glarus.



# NOTES ON SOME OF THE MODES OF PREPARING, BLEACHING, DYEING, AND FINISHING COTTON GOODS, PRACTISED BY LANCASHIRE AND YORKSHIRE DYERS, AND THEIR RESULTS.

BY JAMES SHARP.

IN accepting the kind invitation given me through your worthy chairman, Mr. Ivan Levinstein, to read before you a paper on dyeing, I did not lose sight of the fact that the Manchester Section of the Society of Chemical Industry numbers amongst its members men whose intelligence, combined with the practical knowledge acquired during a long and varied experience in every branch of bleaching, dyeing, and finishing cotton goods, has won for the productions of Lancashire, generally known as Manchester goods, a position in the markets of the world unsurpassed, if even equalled, by that of the productions of any other country. Having thus the great advantage of addressing my remarks to men thoroughly acquainted with these important branches of textile industry, I chose as the subject of this evening's paper that branch in which you are most especially interested, and in which you have had a life-long experience, rather than the special branches of dyeing and finishing mixed goods, which would have afforded me greater scope for comment and illustration; for there can be no doubt, according to my opinion, that the preparing, mordanting, dyeing, and finishing of all the various kinds of mixed goods known as Bradford stuffs, requires a much more extended experience than is needed for the successful treatment of other goods. For many years large quantities of cotton goods have been sent by Manchester merchants to Yorkshire dyers to be dyed and finished, and as a matter of fact the Lancashire and Yorkshire methods of preparing, dyeing, and finishing cotton goods, as well as the machinery employed in



Illustration of dyed Anthragallol: Light brown, mordanted with pure alumina; dark brown, with alumina and iron; light and dark drab, with iron.

position in the molecule. Purpurin is one of the colouring matters from madder. It gives red lakes with alumina and purple lakes with iron. Anthra-



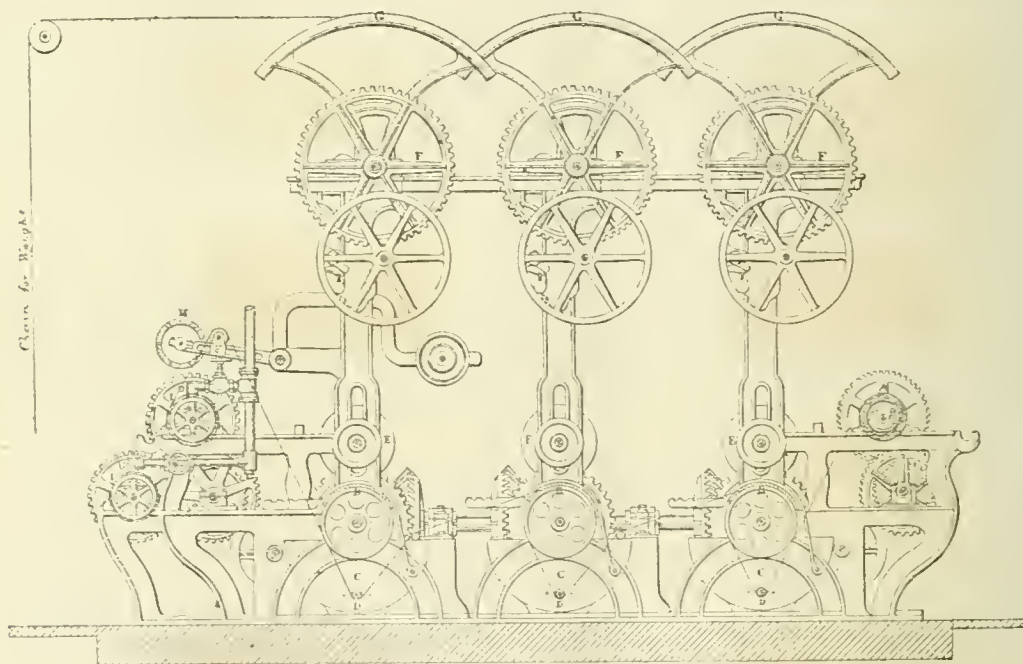
the two localities, are different one from the other, and the results produced are quite distinct. It is to the comparison of these methods, and the results obtained by them, that I invite your attention this evening. In order to bring before you more clearly some of the different methods of treating cotton when mixed with worsted, I shall also have to make some remarks on Bradford stuffs, and shall submit to you some dyed effects in which the worsted has been dyed one colour and the cotton another; but the intention of this will be mainly to show you that in bleaching, mordanting, and dyeing the cotton warp of mixed goods we cannot avail ourselves of the Manchester methods, inasmuch as it is necessary to have a complete control of the application of the dyeing materials.

Before commencing my remarks on dyeing I ought perhaps to say that I consider that the machinery and apparatus employed in Lancashire, such as washers, kiers, scutchers, jigs, pads, mangles, calenders, beetles, and, in short, all other appliances

dyers, and finishers cannot in many respects be surpassed, and having already referred to the high estimation in which the machinery and apparatus of Lancashire are held, I think it only right to admit the fact that on the introduction of the mixed goods known as Bradford stuffs, when no cotton-dyeing appliances existed in Yorkshire, both the Yorkshire dyers and machinists were very much indebted to the great progress which Lancashire had made in the production and application of machinery for dyeing cotton goods, inasmuch as such machinery was applicable to the dyeing, though not to the finishing, of Bradford stuffs. Indeed, some of your machinery was so perfect more than thirty-five years ago, that although many working improvements have since been introduced the principle remains unaltered.

I well recollect the first cotton-dyeing machine that came into Yorkshire—it was a padding machine, supplied by a Manchester firm. At that time the workmen took a keen interest in the introduction of machinery, and on seeing that by this machine goods

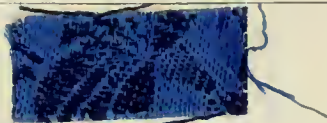
FRONT ELEVATION



TREBLE CRABBING MACHINE WITH PATENT STEAMERS.

used in bleaching, dyeing, and finishing cotton goods, have attained a very high pitch of perfection, and are admirably calculated to produce the results which more especially characterise Manchester goods. The great demand for these goods is the best proof of the high estimation in which they are held by the public. I mention this more particularly because I shall show that in Yorkshire the machinery and mode of treatment differ entirely in several important particulars from those in use in Lancashire, and therefore any special aptitude which the Yorkshire dyers have acquired in the dyeing and finishing of cotton goods, and more particularly in that class known as "Italian cloths," is principally owing to the application of principles distinct from those in use in the Lancashire trade and to the use of machinery capable of producing different results. I do not pretend that the standard of skill amongst the Yorkshire workmen is higher than is the case in Lancashire. An examination of the goods in the Manchester warehouses will furnish abundant proof that the Lancashire bleachers,

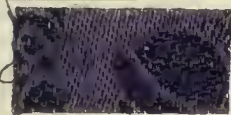
could be so quickly tinted in various shades by simple padding, as it is termed, they named it the galvanising machine. A few months afterwards your dyeing jiggers were first introduced into the Yorkshire dye-houses, and became rapidly adopted throughout the trade. Some years later, viz., in 1852, I became head dyer at the works where the above machinery was first introduced, and for some years after I dyed large quantities of goods with the same machinery. Since that time nearly the whole of the Lancashire cotton-dyeing machinery has been adapted and altered to meet the special requirements of the Bradford trade, by giving to it increased size, increased strength, and additional parts, with the object of making it suitable for dyeing equally with either mixed goods or all-cotton goods in much larger quantities than can be dyed with machinery employed in the dye-houses throughout Lancashire. But what I have to bring before you more particularly is the use of machinery and processes distinct from those employed in Lancashire. Therefore, I propose to show that by treat-



1.—DYED WOOL: Navy Blue, with Indigo and Magenta.  
COTTON: Soluble Blue.



2.—DYED WOOL: Dark Peacock-Green, with Indigo and Picric.  
COTTON: Red, with Vermilline Searlet B.



3.—DYED WOOL: Blue, Indigo.  
COTTON: Violet on Mordant.



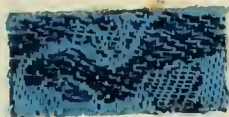
4.—DYED WOOL: With Bichrome and Logwood.  
COTTON: Bleach-White.



5.—DYED WOOL: Dark Prime, with Indigo and Cudbear.  
COTTON: Claret, with Magenta.



6.—DYED WOOL: Dark Olive, Bichrome, Logwood, and Fustic.  
COTTON: Yellow, with Naphthol Yellow.



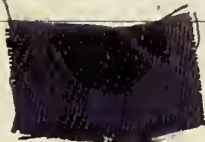
7.—DYED WOOL: Black, with Bichrome and Logwood.  
COTTON: Blue, with Aniline Blue.



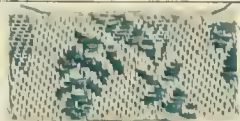
8.—DYED WOOL: Dark Peacock-Green, with Indigo and Picric.  
COTTON: Violet on Mordant.



9.—DYED WOOL: Black, with Bichrome and Logwood.  
COTTON: 3 R. Vermilline Searlet.



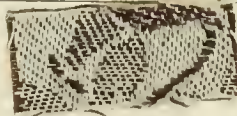
10.—DYED WOOL: Black, with Bichrome and Logwood.  
COTTON: Violet on Mordant.



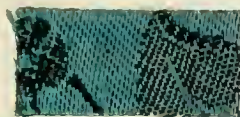
11.—DYED WOOL: Green, with Indigo and Picric Acid.  
COTTON: Bleach-White.



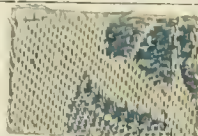
12.—DYED WOOL: Dark Navy, with Indigo and Magenta.  
COTTON: Vermilline Searlet R.



13.—DYED WOOL: With Bichrome and Logwood.  
COTTON: Bleach-White.



14.—DYED WOOL: Black, with Bichrome Liquor.  
COTTON: Green, with Night-Green.



15.—DYED WOOL: Blue, with Indigo.  
COTTON: Bleach-White.



16.—DYED WOOL: Blue, with Indigo.  
COTTON: Magenta on Mordant.



17.—DYED WOOL: Dark Prune, Indigo and Cudbear.  
COTTON: 3 R. Vermilline Searlet.



18.—DYED WOOL: Blue-Black, with Bichrome Liquor.  
COTTON: Magenta on Mordant.



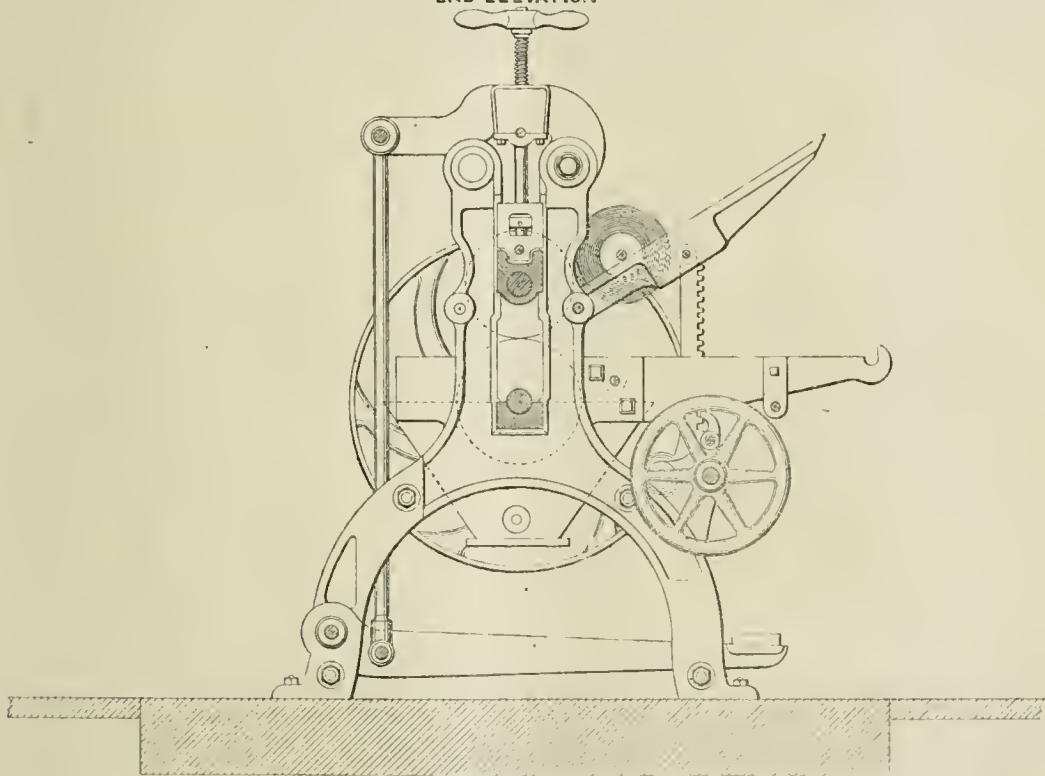
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ing cotton Italian cloths on worsted machinery, the same as if they were worsted (or real) Italians, whether in the processes of preparing, mordanting, dyeing, or finishing, the result is that we approach much nearer to the characteristic finish which especially belongs to the real or worsted Italians than it is possible to arrive at by the modes of treatment generally adopted in the Lancashire dye-houses. The Yorkshire mode of treatment which I am now going to bring before you is more especially applicable to the dyeing and finishing of black and dark-coloured goods. This fact, I believe, has for years been fully admitted both by dyers and merchants in Lancashire, and the result is that the black cotton Italian dyeing has to a very large extent been done in Yorkshire. Although for several years the prices paid for this work were more than double the price paid to-day, no Lancashire dyer took up this branch of the trade, and the present unremunerative prices are owing

Halifax, I am able to place before you drawings of some of their machinery, with all the improvements, some of which are a great advance on the original machine. Simple as this crabbing machine may appear, it is by the skilful use of it in preparing, or as we term it, "gray-finishing," all the various kinds of goods, that we increase the length by two or three yards per piece, produce the gloss or lustre on alpaca goods, give the smooth, close silky finish to Italian cloths or other goods, also impart the soft cloth-handle to all-wool goods and cashmeres, and, what is more important, the finish put upon the goods by this machine is permanent, and withstands the action of the boiling dye-vat. The drawing before you represents a treble crabbing machine, each crab having separate motion or gearing. Practically these crabs are identical one with the other, and each crab has nine parts, used to produce the various finishes. These are marked alphabetically in the order in which they are

END ELEVATION



PADDING MACHINE.

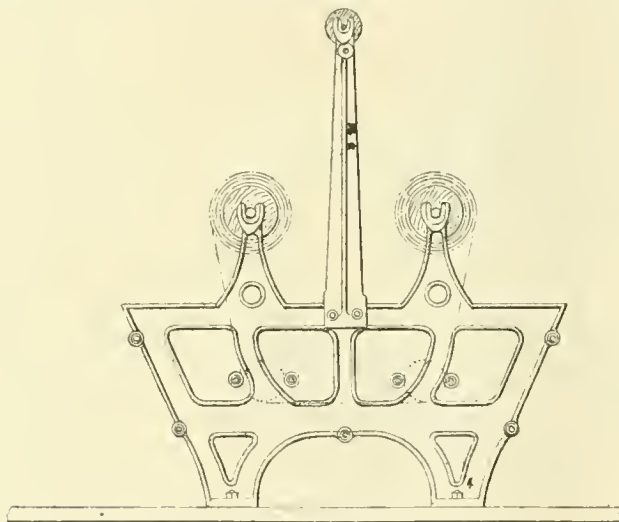
principally to the competition amongst the Yorkshire dyers during the somewhat depressed state of the Bradford trade. The result has been to make the dyeing of cotton goods equally unprofitable with that of Bradford stuff. You may have heard something lately about the advance in prices, of which I will only say that it does not more than cover the increased length and weight of the goods. The prices hitherto ruling had been so low that the invention of both dye and machine makers, as well as the improvements made by the dyer, had failed to make them remunerative.

I will now invite your attention to the drawings representing a treble crabbing machine, which is not used in the Lancashire dye-houses, but is indispensable in the finishing of worsted and mixed goods, and which, as I hope to be able to show you, has some advantages in the finishing of cotton goods. By the courtesy of Messrs. Elkana Hoyle and Sons Limited,

used. A is the beaming or batching roller, B is the brake to put tension on the cloth, C is the trough for boiling alkaline solution, D is the roller in the trough to give increased tension, E is the iron squeezing or crab roller, F is the rack for raising or lowering the top roller, G the lever for putting pressure on the top roller, H the expanding roller to prevent creasing, and I the taking-out or rolling motion. To begin with, the workman causes about five pieces of worsted or cotton Italians to be stitched in one length, with wrappers to protect the outer ends. You will notice that I am passing over the preliminary process of stamping or marking, also of stitching and singeing the goods, as the slight variations in the practice of the different firms do not affect the results which I am desirous of bringing before you. The batch of goods is taken from the singeing and laid in open fold, convenient for being wrapped or batched round the beaming roller A. This only places the goods in a convenient position



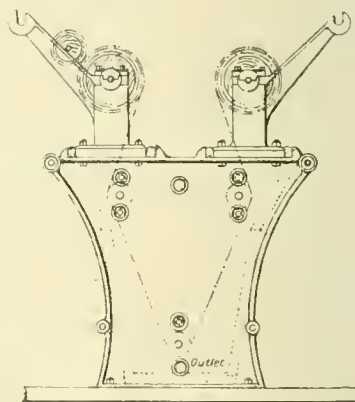
for treatment on the first crab. The trough C is made ready with boiling alkaline solution, when the wrapper which is stitched to the end of the goods is threaded, in open width, through the boiling water under the small roller D, then wrapped round the bottom crab roller E, as shown in the drawing. The brake B is adjusted to give the proper tension to the goods, and the top crab roller E, which has hitherto been raised, is now lowered to bear upon the cloth, and when necessary additional pressure is put upon the cloth by the lever G. The crab is put in motion, and under these conditions the cloth is drawn or made to travel from the beaming roll A through the boiling alkaline solution on to the bottom crab roller E. This process is repeated upon the goods as they are made to travel in like manner to the second and third crabs, after which they are taken on to rollers by the taking-out motion I. The expanding roller E, as already mentioned, is for the purpose of keeping the goods free from creases during the process of batching on to rollers. Worsted goods require to undergo other processes which are not necessary in the case of cotton goods. The steam-

*Side Elevation.*

YORKSHIRE JIGGER.

ing and reversing appliances attached to the crab, and marked J, are only applicable to the finishing of Bradford stuffs, and are the patent rights of Messrs. Elkana Hoyle and Sons, Halifax. This crabbing machine is capable of preparing about 20 pieces per hour, or 200 pieces per day of 10 working hours. I will now submit to you two samples of worsted Italians. The one which is so rough and shrivelled has been dyed without having been crabbed in the way I have described, and affords a practical proof that goods so treated would be completely spoiled. The other pattern, which has a smooth, close, lustrous appearance, and a glove-like handle, has been crabbed previous to being dyed, and this very considerable difference in the result is due to the process of crabbing which I have just explained. Now after placing before you proofs of the remarkable effects produced upon worsted or real Italians by the process of crabbing, I have to point out to you one other result, viz., that in treating cotton Italians with the crab in the same manner as real Italians we obtain an increased length, a smooth, close, even, surface, a softer or more cloth-like handle, and the condition of the goods is not only most suitable for dyeing black, but, what is of very great importance, the weight of the cloth is fully 10 per cent. heavier than that of similar goods which are

prepared by kier bleaching. It may possibly be said that goods are not always kier bleached for black, but as some works do adopt that system I will deal with it in due course. For the present I would just call your attention to the sizing of cotton warps, a process which, as you well know, materially increases the weight of the warp, and it is not necessary for me to tell you that the warp only is subjected to this process before it is woven into the piece. The size being a glutinous body stiffens the threads by coating and filling the capillary tubes of the fibres, thereby making the warp stronger and better to work in dressing and weaving. It also gives a stouter handle to the cloth; and I think I may go so far as to say that the sizing of warps is indispensably necessary, and is not done for the purpose of fraud, whilst the cotton yarn used as weft, and which in weaving is made to form the face of the piece, is kept as light and lofty as possible, because in that condition it gives to the goods a kinder or more cloth-like handle. Now the object of the Yorkshire dyer is to maintain these conditions as far as possible, and we are of opinion that the process of crabbing is best calculated to maintain the original

*Side Elevation*

LANCASHIRE JIGGER.

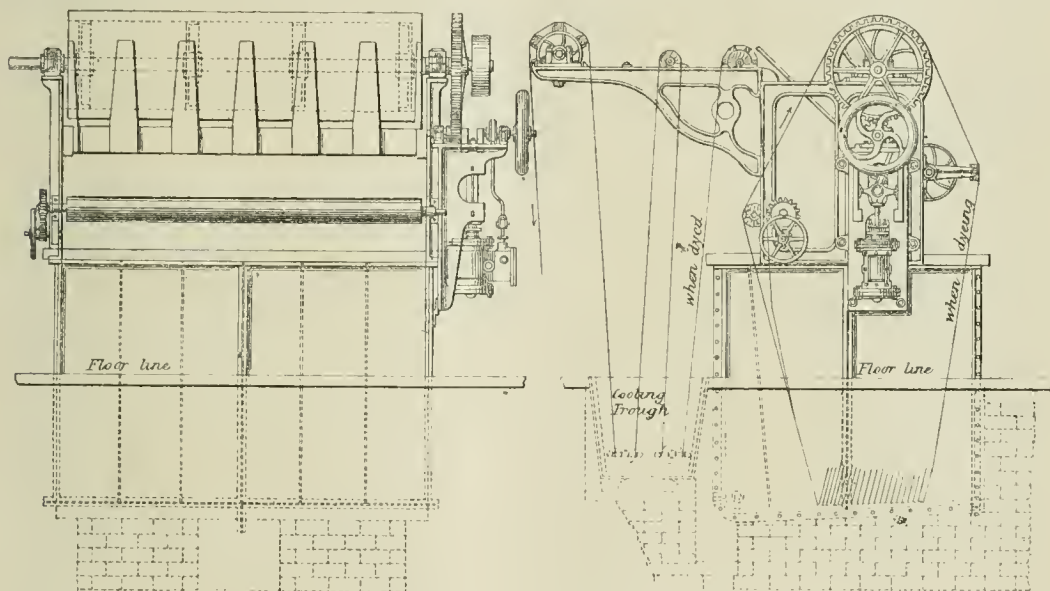
weight of the goods, and to produce increased length and improved handle.

You will now allow me to say a few words on two customs which are prevalent in at least some of the Lancashire dyehouses. One is that when goods are for common black, the dyer receives them direct from the singeing operation, in which case they undergo no preparation equivalent to crabbing. In this case you maintain the weight of the cloth without obtaining any of the other advantages of the process of crabbing. The other is that of treating all goods by the well-known processes belonging to kier-bleaching, quite irrespective of whether they are for white, black, or colours. They are kier-boiled in strong alkali, often under pressure, and subsequently treated with strong acid, then again with bleaching liquid, and afterwards with acids. Now, whilst these processes, which effect a thorough bleaching of the cloth, are quite indispensable when the goods are for white or delicate colours, what are the effects which this severe chemical treatment has produced upon the cloth? It has not merely removed foreign substances, which is necessary in order to produce a pure white, and at the same time made the goods lighter and thinner, but it has also deteriorated the quality of the materials of which the goods are made, and left the latter in a

most unfavourable condition for dyeing a good black.

This brings us to the process of mordanting the goods, and I propose first to explain the processes of mordanting and dyeing as practised in Yorkshire, and in so doing I have to invite your attention to the drawings of two jiggers. The smaller one represents the most improved jigger as used in the Lancashire dyehouses, and as far as regards size it is the same as was first used in Yorkshire. The larger drawing represents the largest size used in Yorkshire dyehouses. A batch or charge for the small jigger is generally 5 pieces of 75yds. each, whilst a batch or charge for the large jigger, as worked in Yorkshire, is 40 pieces of 75yds. each, so that in preparing 40 pieces you employ here 8 jiggers with 4 men, whilst in Yorkshire 40 pieces are prepared on one large jigger with two men to attend to it. I must now direct your attention to the goods, which, after having been crabbed in the very way described, are brought on to these large jiggers, and the first process is to sumac or impregnate the cloth with any of the substances usually employed which are richest in tannin,

dye-bath they are a red shade of black, which is afterwards corrected to the proper shade in various ways, and by the use of a variety of materials. In Lancashire I believe soluble oil is one of the articles much in favour, but it is the method and the result that I have to deal with rather than the dyestuffs and materials employed, inasmuch as results equally good can be attained by the use of very different materials. Therefore, whether you bottom your goods with cutch, sumac, divi-divi, myrabolams, iron salts, copper salts, or by a judicious mixture of some of these, good blacks can be produced, but the comparison I want to bring before you is this, that whilst in Lancashire the processes of preparing and dyeing black are generally done on small jiggers, where the dye is not fixed boiling hot, in Yorkshire the goods are dyed in the large vats represented in the drawings, and in the way which I have described, after which they pass through the usual processes to condition them for finishing, and I believe you are aware that in Yorkshire these goods are finished by hot pressing. This process enables the workman to produce a watered effect on the back and a satin finish



FRONT AND END ELEVATION OF SILK COMPARTMENT BLACKING MACHINE.

after which the goods are saddened, as it is termed, as a rule, with solutions of salts of iron. These processes, as you will be aware, produce a deep slate colour, inclining to black. After this the goods are taken into the dyehouse, where the processes of dyeing are performed with boiling-hot solutions, in the very large dye vats, which are represented in the drawing before us. These vats are made in two sizes. The smaller one is capable of dyeing 40 pieces, and the larger one 80 pieces at once. The drawings will, I think, at once give you an idea of the working principle of the dyeing machines. These vessels are always worked in pairs. In the first the goods are boiled in a solution of bichromate of potash or soda. This process causes the dye to unite firmly with the cloth, and gives a deeper shade of black. In the second vat the goods are dyed with logwood, and in some cases with the addition of fustic. The dye is fixed boiling hot, after which the goods are washed.

Now it is a fact, that in the case of all cotton goods dyed black—no matter what may be the processes or materials employed—when they leave the

on the face side of the goods, like the sample which I now place before you, showing the results attained by the Yorkshire method of preparing dyeing and finishing black cotton Italians. Now the Lancashire method is to finish generally on calenders; and excellent as is the finish produced in that way, it is distinct in every characteristic from that produced by the Yorkshire mode, whilst goods treated by the Lancashire method, in all the processes feel thinner, handle more papery, and, if anyone spends a short time in cutting them up with shears, more especially in the case of coloured goods, he will find the cut is harsh, and gives the idea of dryness and a grating sensation. On the other hand, the Yorkshire-dyed goods feel more kind and clothly, and cut more freely. I think the description I have given will be fully borne out by a careful examination of the two sets of dyed patterns. One set has been dyed in Lancashire, by what I believe is considered one of the largest and best firms, the other set I know to be Yorkshire dyed; and although it is very probable that the quality of the cloths may not be exactly the same, any other differences that exist are more especially due to the



application of distinct principles in the modes of preparing, dyeing, and finishing the goods, in the manner which I have described.

Before concluding my remarks on the dyeing of black cotton Italians, I may refer to the use of fatty matters and alkalis, probably to our mutual instruction. About two years ago three cases of goods were shipped from Manchester, which had been dyed black by a firm whose dyer had either been negligent or inexperienced in this class of work, or else he had employed inferior materials, for although when the goods were delivered to the merchant and examined they were found to all appearance right, and were therefore made up and packed, hermetically sealed, as usual, in tin cases, yet when they arrived at their destination they were covered with a white efflorescent salt, which appeared to have made its way through the whole piece, joining the folds together so as to make each piece a solid mass. Sample pieces were at once sent to this country, where the opinions expressed were extremely conflicting. On examining a sample piece, I attempted to unfold a portion, and found the operation much like skinning a fish, and the white salt, which had a crystalline appearance, turned out to be a mixture of sulphate of soda and fatty matters, partly saponified. I had therefore no hesitation in saying that the fault lay with the dyer, and in pointing out the cause I stated that the dyer had in the last process probably passed the goods through a bath containing some fatty matter, which had been previously mixed with sulphuric acid, and to which had been afterwards added some carbonate of soda,\* and had not afterwards washed the goods out. Therefore on being dried they were strongly impregnated with those substances. Further, that in order to condition the goods for finishing they had undergone a process to make them damp, and this would probably account for the crystals of sulphate of soda, which seemed to have passed all through the cloth, binding the piece as it were into a solid mass. By treating some sound cloth in the manner just described I had no difficulty in producing and reproducing similar effects at will.

There is another point connected with the dyeing of black cotton Italians which is of the greatest importance both to dyers and merchants. I find from some goods that have come under my notice, samples of which I now produce, that some dyers rely almost exclusively, if not entirely, upon copper salts as their mordants. Now, although goods dyed upon such a mordant can be made to present a very satisfactory appearance for a short time, yet when such goods are shipped, as they have been, to distant Eastern markets, the consequences are most serious, for you are aware that the combination between mordant and dye is a very feeble one in the case of such dyes, and you are also aware that logwood, with which all ordinary blacks are dyed, is not itself a fixed dye. Its permanency depends entirely on the application of proper mordants. Now, when copper mordants are relied upon, and more especially if in the last process the dyer uses alkalis or ammoniacal solutions, the copper salts, or otherwise the oxides of copper, continue to exercise an influence on the logwood and fustic (when present), until the black dye is changed into green-olive, for you know that copper salts, logwood, and fustics in proper proportions will dye green-olive on either wool or cotton. But when the evil results from the employ of these articles are aggravated by the presence of materials predisposing the goods to mildew, the consequences become doubly serious and I am able to place before you a sample

of the results arising from such want of experience. I am not aware that I am committing any breach of confidence in stating that the sample which I now place before you represents a rather large quantity of goods, which no doubt would appear to be a satisfactory black when shipped, I am not aware how long ago, but you will probably admit that the pattern, which fairly represents a case of goods reshipped from the East to Manchester is now green-olive and very badly mildewed indeed. In that condition the goods have no value whatever, and the question which I very recently had to decide was, could these goods be restored to good condition. I replied in the affirmative. The one case which was returned as sample (every end of which was like the pattern just exhibited) I have refinished perfectly, like this second short length cut from the goods.

We will now leave the dyeing and finishing of black goods, and I shall say very little on the treatment of coloured cottons, as I intend to bring before you dyed samples of mixed goods, not merely for the purpose of showing you that when cotton is mixed with worsted it has to be bleached, mordanted, and dyed after the worsted has been dyed, and that such processes must be performed in such a manner as not to affect the colour which has been imparted to the worsted, whether we are dyeing the cotton warp the same colour as the worsted or quite a different colour, and it often happens that we cannot employ either the same method of bleaching, the same mordants, or even the same dyes which are admirably adapted for dyeing all-cotton goods. Now, in dyeing cotton goods with colours you have free choice in the use of the materials. You have only to consider what mordants or dyes will enable you to produce the required colour, and it often happens that two dyers, who are equally successful as regards the results obtained, will be using different mordants, different processes, and even in some cases different dyes to produce the same colour. Therefore I do not propose to raise any question as to what is the best method or what are the best materials for exactly matching any given colour on cotton goods, but I will explain the reason why mixed goods would be completely spoiled if in the bleaching of such goods the kier-bleaching process were adopted, and as the stannate of soda and muriates of tin are much relied on as mordants for cotton goods, I will show you that at least in some important processes neither of these materials can be used as mordants for mixed goods. I will first explain the objections to kier-bleaching in the case of mixed goods. A long experience has proved that the worsted must be dyed before the cotton is either bleached or dyed; therefore the use of such materials as milk of lime, soda ash or caustic, or resin soap, all of which are employed in kier-bleaching under pressure, is quite inapplicable to the bleaching of mixed goods, as all these materials are very injurious to wool, and would make the goods quite tender—in fact, if they were applied to mixed goods under pressure, not only would the dye which had been fixed on the worsted be destroyed but the goods themselves would be rendered worthless. And for these reasons the bleaching of the cotton warp in mixed goods is very simple. It is effected by simply working the goods in an ordinary bleaching solution, and then souring off in the usual way. By this mode of bleaching the dye fixed upon the worsted is not injuriously affected.

Now, with regard to the use of stannate of soda and muriate of tin as mordants, I will show that the use of each of these articles would be so injurious to the dye of the worsted in mixed goods as to render their employ quite inadmissible. I have in these three bottles solutions of stannate of soda, muriate

\* Some dyers treat gallipoli oil with sulphuric acid, mixing the two well, and putting this mixture into the finishing bath, after which they add excess of carbonate of soda.

of tin, and nitro-muriate of tin respectively. The three dyed patterns in my hand have been dyed, one sky blue, with sulphate of indigo; one green, with the same dye and picric acid; and one with alkaline blue. I will now divide each of these patterns into three equal parts, and put one portion of each colour into each bottle, and in a very short time we shall see that the colour of the slips introduced into the stannate of soda and the muriate of tin has almost disappeared, while the colour upon those introduced into the nitro-muriate of tin solution remains unchanged. You have thus evidence that in the dyeing of "shot" or two-coloured effects we cannot use the stannates of soda or muriates of tin as mordants. Now the reason for the change in the colours dyed with indigo is that in mordanting with muriate of tin, which is a protoxide, the latter has the power of depriving the indigo-blue of its oxygen, thereby reducing it to the state of white indigo, as you have seen. The indigo itself is not destroyed—its colour can be restored by repeated washing in cold and warm water—but an unfavourable condition has been established on the worsted. Thirty years ago, when shot effects were first introduced, these unfavourable results puzzled most of the dyers in Yorkshire. When cotton-warp goods were first introduced they were worsted-dyed only. The cotton warp was left undyed, for the very good reason that the dyer possessed no proper knowledge of cotton dyeing, or of the materials and processes necessary to be employed—in fact, it was a matter of much surprise to him to find that when cotton-warp mixed goods were put into the dye-bath the worsted only united with the dye, while the cotton remained white. A virtue was made of necessity by putting the goods upon the market in that half-dyed state, when a freak of fashion, ever ready to pursue novelty, even though it presents itself in the shape of ugly and unfinished goods, in this case created a great demand for the goods in question, which were then known and are still remembered as "Parisian figures." The dyed range which I now place before you fairly represents the class of goods then in demand, and they will clearly convey to your minds the fact that when goods have to be dyed "shots," i.e., two distinct colours, the worsted is first dyed one of the colours, leaving the cotton warp white, as in these samples of the old "Parisian figures," and the cotton warp has then to be dyed the other colour, different from that of the worsted. It is in the application of the cotton dye under these conditions that the intelligence of the dyer must bring to bear upon his work an experience that will give him such a command over the dyes as to enable him to fix any dye upon the cotton warp without spoiling the colour which has previously been fixed upon the worsted; and in order to show you how completely this command has been obtained, even in the use of all the new dyes, I have dyed specially the large assortment of two-coloured effects which I now place before you. So far as I know, most of these effects are quite new, and there is some indication that "shot" effects will once more come to the front. I invite your special attention to the clearness of the contrasts and the brilliancy of the dyes. For your guidance a ticket is attached to each pattern explaining the dyes employed. You have here the brilliant scarlets, crimsons, and oranges fixed on the cotton warp, whilst the worsted is dyed black, navy blue, green, green-blue, and olive, and browns, green-blues, and violets are mixed with a great variety of other colours, so that in the dyeing of these "shot" effects I have subjected the application of these dyes to the severest test; and it affords me very great pleasure to be able to add that the dyes I have employed in producing these

effects are made in Lancashire, and are as full in colour, as brilliant in tone, and as reasonable in price as any produced by the Continental makers. The scarlet and crimson dyes, when united with either cotton or wool, have all the brilliancy of cochineal dye, and they possess the great advantage of being easily fixed both upon cotton and wool, without the aid of any of the expensive materials which are indispensable to the use of cochineal, such as bitartrate of potash and tin spirit. You are probably aware that tartar is the mordant for cochineal, and together they dye brown, and it is only by the addition of tin spirit that the rich red colour of the cochineal is produced; that the great beauty of this dye, which has been held in so much esteem, is due to the presence of one of the oxides of tin, whilst the oranges, scarlets, and crimsons of the naphthol series are ready made or substantive dyes. The production of all those new naphthol dyes has been a great boon to the practical dyer, inasmuch as their application has been far more simple than that of the old ones, more especially as regards woollen goods, with which they unite without the use of mordants. I believe I am correct in saying that the fixing of some of these dyes, both on all-cotton cloths and on mixed goods, has been in the hands of some dyers a source of great trouble and anxiety; and it is a fact, that to the inexperienced dyer difficulties do sometimes present themselves which perplex the judgment, irritate the temper, and quite unnerve the man. However, experience proves that nothing is impossible to the mind of man, and that with a little knowledge of chemistry these difficulties may be easily overcome. I do not mean to say that before a dyer can be successful in using these colours he must be a thorough chemist, or even that the scientific chemist engaged in the production of dyes must be a thoroughly practical dyer, but just as the scientific man should possess a greater amount of practical knowledge in the special branches to which he is devoted, so it is equally necessary that the practical dyer who intends to succeed should possess a fair acquaintance with that department of scientific knowledge which is especially applied to his trade. As I have already stated, it always has been and still is a puzzle to many dyers to know which mordants are the most suitable for fixing coal-tar colours. This question is of considerable importance, and we will spend a short time in considering it. The coal-tar colours may be divided into two principal groups—namely, (1) colours with basic properties; (2) colours with acid properties. The colours belonging to the second group are frequently derived by combining those of the first group with sulphuric acid. The following are the names of some of the principal coal-tar colours divided into two groups just named—*Basic Colours*: Magenta, saffranin, chrysoidine, Bismarck brown, malachite green, brilliant green, aniline blues soluble in spirit, methyl violets, methylene blue, etc. *Acid Colours*: Eosines and other resorcin colours, roccelin, acid magenta (sulphorosaniline), naphthol scarlets, naphthol oranges, naphthalene yellow, sulphomalachite green, blues soluble in water, etc. Colours which possess basic properties have, as a rule, much greater affinity for cotton than those possessing acid properties. The basic colours will dye cotton either with or without a mordant, but as a rule it is preferable to use mordants for these dyes. On the other hand, colours with acid properties do not unite with and dye firmly cotton goods either with or without a mordant, the combination between the two being so feeble that these dyes may be considered, so far as regards cotton dyeing, simply as impregnation colours. Mordants may thus be entirely dispensed with in using these acid colours,



and I consider the use of mordants with them as being, with perhaps a few exceptions, an absolute waste of time and material, and in many cases injurious to the colours. There is only one rational mode of dyeing, or perhaps we may more appropriately say painting, cotton with these colours, and that is by impregnating the cottons with a strong solution of the dye, and then drying the goods without washing, etc. The effects which I have shown you this evening on shots or single-dyed cottons have in every instance, where colours belonging to the second group have been used, been produced without the assistance of any mordant whatever. I think I have now trespassed sufficiently on your valuable time, and I beg to thank you for the indulgence you have shown me in listening so patiently and attentively to my paper.

#### DISCUSSION ON DR. BOURCART'S AND MR. SHARP'S PAPERS.

The CHAIRMAN: We are much indebted to Dr. Bourcart for the pretty exhibition of prints from Alsace and Switzerland. These prints are specialities, and differ from our average prints in style as well as in execution, but on account of their high price and their special character the demand for them is naturally limited. We also have to thank Dr. Bourcart for bringing before us the dyeing properties of anthragalol. This colouring matter, if yet excluded from competing with alizarin, may at some future day be a valuable addition to our dyewares, and it must certainly be interesting to our members to have their attention called to new products which at some future time may become of practical importance. We have also listened with great attention to Mr. Sharp's valuable paper, to whom great credit is due for kindly coming forward with the object of enlightening our minds on a subject which is shrouded in a good deal of apparent mystery. Such a paper as we have just heard must conduce to the placing of the art of dyeing on a much healthier basis, for after all there do not exist so many real secrets in this art, and Mr. Sharp's attempt to put certain matters connected with it on a more scientific foundation is a most praiseworthy one, and the sooner dyers follow this course the quicker will be their progress. Dyers as a body are very conservative in their trade secrets, but the days of secrecy and mystery are passed, and the success of a dyer depends to-day far more on his personal skill and knowledge than on the secrets or receipts inherited from his father and grandfather. Mr. Sharp rightly points out that successful dyers need not necessarily be great chemists, but they ought at least to understand that part of chemical science which has a direct bearing on their trade. Mr. Sharp has shown us several advantages of the Yorkshire dyers, and the superiority of some of the machinery used in the Yorkshire dye-houses; but considering that the art of cotton dyeing is much older in Lancashire, I may rightly presume that in some specialities our dyers excel, and I hope that before long some Lancashire dyer will set us right on this point by reading a paper on Lancashire dyeing specialities. Considering also the very large number of gentlemen who are present this evening, who are thoroughly acquainted with the subject of the paper, I hope that we may get some information in course of the discussion about the mordants best adapted for coal-tar colours. Many dyers only know sumac and tin (muriate of tin or stannate of soda) as being suitable for the purpose, and whatever colours they have to dye, irrespective of their composition and their special character, sumac and tin, for good and evil, are resorted to. The sizing of cotton warps mentioned by Mr. Sharp may also induce some gentlemen to give us the benefit of their opinion; and surely

this is a subject of which we ought to know more in Lancashire than our friends do in Yorkshire, and it will be very interesting to know to what degree the process of sizing is legitimate, for we all know that it is absolutely necessary for manufacturing purposes to put a certain amount of size on the warps. There are several other points in Mr. Sharp's paper which will suggest themselves to you as practical men, and I have now the pleasure of inviting you to give us your opinions on the subject which Mr. Sharp has so ably treated.

Mr. J. CARTER-BELL: It appears to me, from what we have heard from Mr. Sharp, that the advantages which the Yorkshire dyers possess over the Lancashire dyers in the dyeing and finishing of special cotton goods, such as Italian cloth, etc., principally are due to different machinery, and Mr. Sharp, perhaps, will tell us why the treble crabbing machine has not been generally introduced into Lancashire dye-houses.

Mr. HART: Is it the action of the copper which causes mildew, and does it affect the colour only?

Mr. CUNLIFFE: May I ask by what process you restored the colour and afterwards dyed the goods in the ordinary way?

Mr. SIEBOLD: I should like to know whether the damage to the cloth has been proved to be caused by mildew?

Mr. GEORGE E. DAVIS: I have been much interested in Mr. Sharp's paper, especially that portion relating to sizing and mildew, to the study of which I have devoted considerable time. By the appearance of the damaged goods upon the table I feel much inclined to say that the discolorations do not proceed from mildew but from quite another cause. At one time every obscure stain was ascribed to mildew, and by the uninitiated even yet, but I hope the time will soon be past when this state of things is possible. One point struck me during the reading of the paper, and that was the fact of the adhesion of the various folds of damaged cloth. When we come to consider that there was a large amount of sulphate of soda present, it may readily be seen how this took place. Sulphate of soda crystallises with much water, and the crystals melt at a rather low temperature. Under the excessive heat of the tropics it is possible that the crystals were melted, and afterwards recrystallising bound the whole mass together as one piece. I did not understand from Mr. Sharp's paper whether the mixed goods dyed with aniline products were to be considered as fast dyed goods, so far as the cotton is concerned.

Dr. J. GROSSMANN: I am very glad that Mr. Davis has led the discussion into a different path. The question of mordants is a very important one. As Mr. Sharp has admitted these colours as they are at present, are not fast, and it seems to me that there is no mordant known which will fix them, it is a question which requires to be worked out. There are several experiments which have been introduced and which promise to be of some use, and I am very sorry that Mr. Sharp has not made a few remarks upon the new mordants. There are, for instance, the sulphur mordants for fixing aniline colours, and it would have been very interesting to know what effect they have on aniline colours, and how far they improve the fixing of them. I understood Mr. Sharp to say that to a certain extent bleaching deteriorated the goods, but I think that careful bleaching will not deteriorate them. The new way of bleaching by chlorine, if conducted too quickly, is injurious, but if carried out in a right way it is successful. Of course it is a question of time and money. I have noticed that boiling with chromates in dyeing is another process. There are still a great many mordants used for which there

is no necessity—the chromic acid itself, except when it acts with gelatine and so forth. The chromates are used not in the state of chromic acid but as chromic oxide, therefore why not use these mordants in the form of the preparations to which they have to be brought after all. Why should dyers use bichromates? Why not use chromate salts at once?

Mr. GRIMSHAW: The question of mordants appears to me to be the most important chemical aspect of the subject of dyeing. The object of dyeing, I take it, is to place upon the fibre a colour as brilliant and as satisfactory as possible, at the same time as fast as possible. We are all aware that colours will take more kindly and more firmly to animal fibres such as wool than they do to cotton, and this I think is partly due to the chemical nature of the two fibres. There are two ways of dealing with these fibres, one is to treat them in such a manner that by some means you alter the structure of the fibre, and the other way of mordanting these fibres is to cause the dye in a soluble form to combine with the mordant which has been previously placed upon it, and it seems to me that a mode of fixing them could be arrived at by the processes described by Mr. Sharp. Accepting, then, as a fact that one of the objects is to fix these colours upon fibres, I cannot help myself always regretting that so many of the new dyes that are brought before us are, I may say, in quality the opposite of the age in which we live—that is to say, the advantages are rather fast but the dyes are not fast at all. I think we have to thank the manufacturer of aniline dyes for furnishing us with such excellent and brilliant colours that are less costly, and the supply of which is practically inexhaustible, and I do hope that the chemists and dyers making these colours will see that they stick in the cloth.

Mr. SHARP: In reply to Mr. Carter-Bell, I must confess that it is rather difficult to state the reasons why the Lancashire dyers have not adopted the treble crabbing machine. I suppose the Lancashire dyers and bleachers have a method of treating cotton goods which has met with great success, and by this method they have been able to dispense in the dyeing and finishing of some cotton goods with this machine; but then, in the dyeing and finishing black dyed Italian cloth the treble crabbing machine and the Yorkshire mode of dyeing is in my opinion superior, and therefore that branch of dyeing has come to Yorkshire; and to give you a practical illustration, I may tell you that for years 2d. per yard was paid for dyeing these goods black—to-day considerably less than 1d. per yard is paid, showing that it was not the want of profit which kept the Lancashire dyers from taking this class of work. I do not think for one moment, in reference to Mr. Hart's query, that copper salt produced the mildew. I rather should think the contrary. What I meant to say was that some dyers seem to entirely rely on copper salts as mordants for black, and when the goods are shipped to distant markets the copper salt, or probably the copper oxide, continues its action on the black dye, and ultimately changes it into a green or olive. I do not think that Mr. Cunliffe's question comes within the scope of my paper, and acting on the advice of the chairman I decline to answer the same. Mr. Siebold desires to know if the damage to the cloth shown to you has been proved to be caused by mildew. I must tell him that I have not microscopically examined the cloth, but from its appearance I would judge the red spots to be due to mildew. It is remarkable that those parts of the cloth which were nearest to the boards of the cases were worse spotted than the other parts. The principal question which I had really to decide was if these goods could be restored to their original colour; and as I

have shown to you I succeeded in this, and I have no doubt in my mind that the change of colour from black to olive was caused by the excessive use of copper salt used as a mordant. With regard to the use of bichromates mentioned by Dr. Grossmann, I must say that dyers have to use that which is offered to them by the chemists; and although I am not prepared to deny the probability of the chromates being cheaper, I do believe that at the present exceedingly low prices of bichromates it will be difficult to get anything cheaper. It is quite true that the injury caused to the cotton by bleaching entirely is a question of degree, and the longer you treat the cloth and the stronger the bleach the more injurious is the effect, and *vice versa*.

## Newcastle Section.

The election of officers for next session took place, when the following gentlemen were elected as officers for Session 1881-5:—

Chairman: J. C. Stevenson, M.P.

Vice-Chairman: B. S. Proctor.

Secretary and Treasurer: J. T. Dunn, M.Sc.

Auditor: N. H. Martin.

### Committee:

P. P. Bedson, D.Sc.

G. T. France.

C. H. Gunningham.

John Glover.

A. S. Herschel, M.A.

John Morrison.

John Pattinson.

H. R. Proctor.

W. W. Proctor.

J. F. Stark.

T. W. Stuart.

The discussion on Mr. H. Proctor's paper on "Improvements in the Methods of Estimating Tannins," and on Mr. Gunningham's paper on "Improvements in Sprengel Air Pumps," was adjourned until the next meeting of the Section.

## Glasgow and West of Scotland Section.

Chairman: Ed. C. C. Stanford.

Vice-chairman: Professor Ferguson.

### Committee:

George Beilby.

Professor Crum Brown.

J. Christie.

Dr. John Clark.

Professor Dittmar.

George G. Henderson.

George M'Roberts.

James Mactear.

Professor Mills.

James Napier.

T. L. Patterson, jun.

J. B. Readman.

Dr. Edmund Ronalds.

F. J. Rowan.

R. R. Tatlock.

Dr. William Wallace.

### Hon. Treasurer:

J. J. Coleman, 45, West Nile Street, Glasgow.

### Hon. Secretary:

Dr. James J. Dobbie, Chemical Laboratory, University of Glasgow.

### MEETINGS FOR 1881.

APRIL 8TH.—(1) On Algin. By Mr. Stanford. (2) On some Points in the Technology of Oil of Vitriol. By Mr. Mactear. (3) On Nickel Vessels for Chemical Operations. By Professor Dittmar.

APRIL 20TH. (Special meeting)—Discussion on International Methods of Testing and Sampling. Introduced by Mr. Tatlock.

MAY 13TH.—(1) On Different Methods of Producing Cold Artificially. By Mr. Coleman. (2) On the Composition of Tobacco. By Dr. Clark.

## INAUGURAL ADDRESS BY THE CHAIRMAN,

ED. C. C. STANFORD, ESQ.

WHEN the British Association met at Dublin in 1878, a well-known professor there, who combines the highest scientific attainments with a delightful sense of humour, and who takes a fatherly interest in the local Zoological Society, undertook to introduce the noble animals to the British Association. He afterwards performed the much more difficult task of introducing the British Association to the noble



animals. The result of the latter introduction was highly disappointing—the noble animals were not at all impressed with the dignity of their human types. He has assured us that a vast amount of eloquence was necessary to convince the lions that there was anything remarkable or worthy of imitation about any of the great scientists. One old lion is said to have remarked (of course in his own language) that, in his opinion, they were a very average lot, that their looks were nothing to boast of, and that they talked a vast quantity of twaddle. The appearance of the president did not frighten him, and it is said that he even went so far as to remark that although he were a storehouse of valuable and extensive information, he thought if left alone with him for a little while he could acquire and digest at any rate the substance of it. Now, there is no doubt the lions have some noble qualities in which scientific man is deficient. One of these is the heaven-born gift of silence—they do not talk. My position this evening is somewhat like that of my friend the professor—I have to display the earthly gift of talking; you, on this occasion only, to reflect, the heavenly gift of silence. That, however, is your fault and not mine; and it is some comfort to me to reflect, that however much you may be dissatisfied with the medium of introduction, you are not allowed, again, on this occasion only, to criticise too severely his parts of speech. You have accorded me the happy privilege of presiding over the first session of the first Scotch Section of the Society of Chemical Industry. It is my pleasant duty, therefore, to introduce that Society to you, and my still more pleasant duty to introduce you to that Society. I have this great advantage over my friend the professor, that however much his newly-made acquaintances might mutually benefit each other at a distance, a too intimate mixture would be scarcely desirable. There might be a good deal of decomposition, and a considerable rearrangement of atoms and molecules, perhaps tending to an increase of carnivora at the expense of science. In our case it is different, for I am certain that the more intimate we become with our English friends the more we shall like one another, and that every atom we add here to the chemical industrial molecule will so strengthen it that very considerable outside forces will be unable to decompose or even to discompose it.

The Society of Chemical Industry was only established in June, 1881. It is not yet, therefore, three years of age, but in that short period it has risen to a membership of over 1,500, and it contains among its members chemists and others engaged in every variety of work in any way connected with the science. The Society publishes a most valuable monthly journal, the excellence of which must be admitted by all who peruse it, containing as it does not only the papers read before the Society, or its sections, but also a complete abstract of papers, communications, and patents relating to technical chemistry. The main object of the Society is to bring together, by personal contact where possible, and for interchange of ideas, those connected with any branch of industry requiring chemical knowledge. This, in a broad general sense, makes the Society extremely comprehensive. Scarcely a manufacture exists which does not at some point or other touch the science of chemistry, and every year adds to the number of manufacturers who find it advisable, and indeed necessary, to consult or employ a chemist. Another object of the Society is to promote good fellowship amongst chemists of all denominations; and I claim for it that it has already made wonderful progress in bringing a class of men together who have been hitherto rather inclined to hold aloof from one another. I speak particularly of large chemical manufacturers.

The meetings of this Society have shown that there is very much to be gained and nothing to be lost by the discussion of the faults of old processes and the advantages of new ones. These meetings have already resulted in some unity of action on common grounds, and this tendency is sure to increase. There is also a great advantage to manufacturers to meet professional, analytical, and other chemists engaged in widely different occupations, because those who make one large chemical are very apt to run in a groove, and not think of a manifest improvement which may at once occur to a skilled outsider. We all know how long the railway engineers insisted in running on their unjointed rails before they could be made to believe in the practical application, much less the advantage, of the coachmaker's celebrated fish joint, and which they deem now an absolute necessity. With the object of insuring good discussions on a wide basis, the Society has always welcomed strangers at the meetings, and its members are glad to hear any who can throw a side light upon the subject, knowing as we well do that these side lights are often the best to bring out the shadows. The annual general meetings of the Society, held in London and Manchester, have done much to promote intercourse among chemists, and no doubt the next to be held this year, in Newcastle, will be equally successful. The Society has already sections in London, Liverpool, Manchester, Newcastle, and Birmingham, and we are met together this evening to inaugurate the last, and I hope not to be the least, that for Glasgow and the West of Scotland. The number of members in Scotland are now 158. Of these 109 have joined this Section, and over 50 have been added within the last month through the officers of this Section. A rough analysis of these numbers shows the members to be made up as follows: Professors and demonstrators of chemistry, 15; analytical chemists, 25; manufacturing chemists, 69; chemical engineers, 8; chemical merchants, 8; chemists engaged in dyeing and printing, 10; chemists engaged in iron and steel, 5; chemists engaged in oils and soaps, 9; pharmaceutical chemists, 3; metallurgists, 2; chemical apparatus maker, 1; alkali inspector, 1; doctor of medicine, 1; total, 158. Further analysis of the manufacturing chemists would again divide their manufactures into that of almost every known chemical product from sugar to dynamite. We like to manage our own affairs in Scotland, although, with all our agitation, we have not yet got our Secretary of State. With our Section, however, no agitation is necessary—the General Council in London is anxious to give us perfect liberty, and we have therefore not only a secretary but a committee of our own; and I have no doubt they will be glad to find at head-quarters that we are self-supporting. There is little need to introduce our committee there, for many names in it are known far beyond Scotland; and I think all will admit that they are a thoroughly representative body, and that they give an earnest that we shall do some valuable work. Two of our most distinguished Edinburgh chemists have joined the committee. We have endeavoured to fairly balance professional and practical men, and I may truly say that with us honours are divided. It is only fair, however, to you in this introduction to remark that the Scottish lions, though generally reticent, have always spoken to some purpose on chemical and other scientific subjects.

The Chemical Section of the Philosophical Society of Glasgow has been established over fifteen years, and during that period has enriched the journal of that society with a number of valuable communications, and added many a page to the *Chemical News*. It has been instrumental, also, in preserving the memory of one of our greatest native chemists,

the illustrious Graham, by the establishment of the Graham Fund. This fund is intended to afford a Graham lecture and a Graham medal every three years. The first lecture was given three years ago by Professor Chandler Roberts, Graham's successor at the Royal Mint. The second will be given on the 20th of this month by Dr. R. Angus Smith, whose personal reminiscences of Graham will be very interesting. We are proud to know that the first medal awarded for research in chemical technology, although open to all, was presented to our distinguished colleague, Mr. James Maclear, for his researches in chemical technology. We may also lay claim to a most extensive range of chemical manufactures in our district. I believe almost every manufacture is represented, and some which are unknown elsewhere. I was informed by one who had seen all the great exhibitions of chemicals yet shown throughout the world, that our collection got up when the British Association visited Glasgow, and much of which is still in the Kelvin grove Museum, was the finest ever brought together. I need only point to our programme for the short session we have before us to prove that we have no intention of furnishing blank sheets as our contribution to the journal. On the contrary, we shall have some difficulty in getting through our papers in the time. We have therefore all the elements of success, and if the new Section is not successful we shall have ourselves to blame.

As a prelude to the work of the session, I shall not attempt to pass in review or even to sum up the numerous discoveries which have been recently made in chemical industry: to do so would be to repeat our journal, so well has it kept pace with the times. Probably there is no science which has made such gigantic strides during the last fifty years as that of chemistry. If you take down a good encyclopedia of that date, you will be surprised at the small place allotted to a science the boundaries of which are so extensive now. In the Encyclopedia Britannica of 1830, Brewster's edition, the three alkalis, potash, soda, and ammonia, occupy little more than a page. They are described under the names of the vegetable, the mineral, and the volatile alkali, and it is curious to read, so late as this, of the recent discovery of the metals potassium and sodium by Mr. Davy, and Brewster's remarks thereon. He says: "These results, not yet sufficiently familiarised, and at war with all our late anticipations of the nature and composition of the alkalis, still surprise us." It is curious to read in the same work that soapmakers were then indebted to kelp or barilla for their supply of soda, and of this, kelp only contained 2 to 5 per cent. The importance of analysis is alluded to in the statement that for want of it "some buyers gave £8 per ton for kelp containing 4½ per cent. of soda, and £11 11s. for kelp containing less than 1 per cent. of soda," but the soda must have cost the soapmakers then what would be equal to £80 to £100 per ton for soda ash. A vast change has come over our country since those days. The annual value of the production of these three alkalis is now counted by millions of pounds sterling—the Leblanc, and later the soda ammonia process, for soda; the Stassfurt mines for potash; and the gasworks, and soon, probably, all works burning coal, for ammonia. Anyone who left the earth fifty years ago would now find everything touched by the busy hand of man completely changed. He would have to go into the country and hear the song of the lark, and see the blush of the rose, to find anything familiar to him. Fifteen years, however, before this date, there were chemical works here. The Messrs. Turnbull, of Camlachie, were making wood products. Acetate of lead was then £112 per ton, acetic acid and naphtha 10s. a gallon, charcoal £7 per ton, and Prussian blue 5s.

per pound. They also made bichrome in 1820—it was then sold at 20s. per pound. Some of the fluctuations in prices have been very extraordinary, but in nearly all the tendency has been to largely diminish cost of production and price, and some manufactures have been completely revolutionised by the discovery of new sources of raw material—for instance, the Stassfurt mineral, reducing the price of muriate of potash from £27 per ton to £7 per ton, and bromine from 40s. to 1s. 3d. per pound; the Peruvian caliche, reducing iodine from 40s. to 5s. per pound; and now we read of that wonderful country, California, having mountain ranges of pure alum and pure sulphur. It is rather difficult to compete with such titanic factories. Pleasant, however, as it is to look back and mark the gigantic strides of fifty years, I would rather take courage and look forward; and however much we may congratulate our countrymen for their large share in the great discoveries which mark that advance, it may be more healthful and more politic to see if we are really still ahead in the race. We know that even where as a nation we have been the pioneers we have allowed other nations to outstrip us—for instance, in our boasted postal communication, which in some outlying parts of our island is wretched, and we have not yet the halfpenny letter nor the sixpenny telegram. It is quite the popular fashion now to believe in the power of chemistry to achieve great things—everybody believes in it in a general sort of way. But do we really believe in it? and if so, where is the evidence of our faith? What do we do to extend its boundaries, to increase our knowledge—in short, how do we encourage original research? Does our government believe in it? if so, how much? We all await with impatient interest the report of the Royal Commission on Technical Education, in which our first president, Prof. Rosecoe, is so energetically working. Does anyone doubt, however, that the report will be sadly against our negligence in this direction? When we read of a magnificent laboratory for analytical and technical chemistry of great architectural beauty and of splendid internal arrangement, to be erected at a cost of £70,000, we know that it is not for us, it cannot be in this wealthy country—we must go to a much smaller, a much poorer, but a more intelligently governed country for that. I allude to the new laboratory in course of erection at Zurich by the Government of Switzerland.

If you talk to John Bull about the importance of setting chemists to work to verify the atomic weights, you will find an impatient man with his pockets closely buttoned; but if you propose to him a process, however absurd, by which quicksilver can be made into real silver, or by which a gold sovereign can be easily split up into ten silver half crowns, you will secure an open-mouthed listener; indeed, if the process be plausibly described and thoroughly impossible, you may perhaps put your hands in his pockets and turn them inside out—at least it has been done over and over again. We have only just begun as a nation to teach our people to read and write, and it is only quite recently that we have formulated any laws against ignorance. As for anything higher, while other nations have had excellent higher-class and technical schools, we are only beginning to think about these. But we are at last waking up, and no doubt when we do set about it, as usual, we shall not underdo these. Scotland is to be still ahead, for only last week the Peterhead fishermen preferred a request to the Treasury, not for better nets, not for safer harbours, not for finer piers, all of which they want, but for further facilities to aid scientific inquiry into the habits of the herring. I do not know how many thousands of years



this most ancient of industries has been carried on, but it is the occupation on which a large portion of our population live, and it is refreshing and encouraging to find that at last they themselves think it advisable to know something of the habits of the fish they want to catch. Probably, however, the Government will have no funds available for such trifles. The fact is we must have compulsory laws to teach our people. Our Education Act would be little use if it were permissive and confined to those who wanted to learn. According to the *Pall Mall Gazette* France has 30,000 school libraries and 4,000 free public libraries. We have only 96 free public libraries. Even in this city, where private benevolence has done so much in the provision of free public libraries, we cannot get the Act adopted to house the books properly, and probably we never shall as long as it is permissive. Rates for educational purposes are sure to have a marvellous reflex action upon the habits and intelligence of our people, and will pay them a hundred and a thousand fold. Our people have a coarser, rougher way of enjoying themselves. Our annual drink bill amounts to £120,000,000. It is painful to reflect what an amount of rational enjoyment might be got out of even a small proportion of this expenditure, if it went to museums, colleges, libraries, and other public sources of intellectual amusement and instruction; yet most of this sum is absolutely within the power of the people to spend as they like. Every employer of labour in this district knows what the vacant Monday after the pay means to himself and his men, and that higher wages do not always imply greater comfort or enjoyment to the workmen. Do we, as chemical manufacturers, really believe in original research? and if so, how much? Where is the evidence of our faith? What do we do towards verifying the atomic weights on which our products are bought and sold, and on which all our manufactures are based? In one of our leading shipyards there is what is called a scientific department, with Froude's tank erected at great cost, with all necessary apparatus and a scientific staff. It is quite distinct from the general work, and devoted entirely to experiment. If that be advisable and necessary in mechanical work, how much more is it necessary in chemical manufactures? But where is the large chemical works that has such a scientific department? Where shall we find a laboratory which has over its portals without "Original research," and which has for its motto within "Experiment, experiment, experiment"? Where shall we find a laboratory which does for any of the chemical manufactures what the Rothamstead laboratory has done for agriculture? The splendid series of researches which have adorned the proceedings of the Royal Society for so many years, and rendered the names of Lawes and Gilbert famous, prove the value of that laboratory. The honour paid to Sir J. B. Lawes shows that even this country can recognise the value of such work—after it is done; and I am very glad to see that Dr. Gilbert has just been appointed Professor of Rural Economy at the University of Oxford—rural economy being Oxford for agriculture; and it is a Greek culture of rather a novel kind for that great University, and shows an awaking more and more to the claims of science. The idea of a chemist at Woolwich was considered extravagant and unnecessary at one time, but the large scientific establishment now required there shows that in the view of our practical nation it has paid its expenses over and over again. The highly-deserved honour bestowed on its distinguished head, our late President, Sir Frederick Abel, shows again that our nation can appreciate the value of such a great work—again after it is done. We are all wise

after the fact. But are we really convinced that a scientific department is absolutely necessary in manufacturing industry—that is, to us, will it pay? If we profit by past experience, it will certainly form an important feature in the ideal chemical work of the future. Let me illustrate what I mean by a reference to any works, say to gasworks. Of course where other things are equal, the work will pay best where the greatest attention is paid to fulness of output, to economy of labour, of fuel, and of general expenses; but one manager might occupy much time in seeking the cheapest method of getting rid of his waste products. He may have made an excellent contract extending over a term of years for their disposal, only to find out afterwards that he has lost the raw material for a valuable series of new products. I will assume that another gasworks had chemists constantly working on new ground, and that they had discovered the value of the ammonia and the tar which the other was seeking to get rid of as a nuisance. The chemists might or might not have discovered the coal-tar colours, but they would certainly have utilised the ammonia and tar, and it would not have been a subject of discussion now as to whether the lime should be put in the retort instead of the purifier. There can be no doubt as to which of these works would pay best. This is without in the least undervaluing those actual business habits which succeed everywhere in everything. Such instances might be indefinitely multiplied, and the moral is, Don't starve your laboratory; for while you are building a large factory on the usual approved principles for making some marketable product, full of devices for labour and fuel economy, some pale-faced student in a far-off foreign laboratory may be patiently, quietly, but surely undermining the whole fabric.

Now, assuming its value how is this original research to be taught? There is one thing we can never teach, and that is originality. According to Carlyle our population consists of so many millions, "mostly fools." It is bold to differ from such an authority, but this expression has always appeared to me a rather unfair and indirect hit at the fair sex, who form the majority, and the representative of that sex that he knew best was by no means a fool. If, however, the observation ran as so many millions "mostly level," many would endorse it. Nature has always a great tendency to return to a common pattern, and a man whose stature is head and shoulders above his fellows is a marked man all his life; nothing in his surroundings suit him—they all seem to be made for somebody else. So it is with the mind, you cannot crib and confine a great mind, you cannot cram it into the small places of other men any better than you can squeeze a man into a baby chair—its originality overflows and impresses itself on all he says and does. Such men will always be scarce: they mark epochs of thought. Genius may be an infinite capacity for taking pains, and many may attain to it; but a Faraday and a Siemens are men of a generation. No education that we can devise will create such men, and if it is genius in others it is inspiration in them. In fact, it is most probable that our method of education by pass examinations may have a directly opposite tendency. It will certainly level up the majority of the masses, but it may level down the minority who ought to go up higher. Our educational machine, which cuts all its intellectual straws the same length, has an undoubted tendency to lop off the big heads of the long ones. Professional crammers have proved that "an egg shell may be made to look very like an egg." Original research, then, like exploration, is a

very difficult thing to teach. It is easy enough to go along a road that is well lighted, especially if you know the way, but when you come to the end of it, and have to explore a new territory with little light to guide you, then the difficulty commences. The usual experience is, although our chemical schools are rendering it easier, to get young chemists for all ordinary work. Those who have the talent for original investigation are scarce. And who is to teach it? Our professors say they are too much occupied with their present classes; the analytical chemists say they are too much engaged in analytical work, and we the manufacturers in practical applications; but all can assist, and a perfect study of each separate branch is necessary. Original research demands the highest theoretical knowledge of chemistry, the most intimate acquaintance with the best analytical methods, and for use on the large scale the most thorough practical knowledge of the branch known as chemical engineering. The three talents are different, and are not often found to perfection in the same individual. I am doubtful if any technical college could teach this important branch of chemical engineering, and it is here particularly where practice in a chemical works is necessary. In dealing with the wide range of substances, with such various properties, we require a knowledge of strength and properties of materials which is quite special. Platinum, silver, nickel, copper, lead, antimony, zinc, iron, and other metals are all pressed into our service. Vessels of stone, brick, porcelain, lead, and iron of all sorts and shapes are employed. Even our artisans must be the best that can be obtained. The ordinary plumber with his soldering iron is no use to us. It takes a bricklayer with a head on his shoulders to build our intricate furnaces. Even our mason must be able to work the hardest of stone. It is no ordinary kind of engineer that we require when we have to pump such liquids as oil of vitriol, hydrochloric acid, and caustic soda, or exhaust such gases as chlorine, sulphuretted hydrogen, sulphurous and carbonic acids. All this means a pretty wide range of experience, and one impossible to learn theoretically. The advantage of a scientific laboratory in a chemical works is that this knowledge of materials is close at hand, and nowhere else could the great difficulties in the application of new knowledge to new manufactures be so easily worked out. We all know that some reactions which succeed in the laboratory are impossible on the large scale. On the other hand, it does sometimes happen that difficulties in the laboratory disappear in the works. Take, for instance, the ease with which large quantities of chlorine are manipulated without any escape, and the many instances in which the influence of large masses of liquid or solids assist reactions. Great as was our President's discovery of the Weldon process it seems to me the enormous difficulties, patiently overcome in the invention of the necessary apparatus to work it on a large scale, make the discovery far greater. The actual discovery of the Bessemer iron process, which has so revolutionised the iron industry, and which is a strictly chemical process, appears to me to bear only part of the merit due to the wonderful mechanical contrivances invented to work the process on such an enormous scale by that talented inventor.

It appears to me that what our schools have mostly to aim at is the widest possible teaching of chemical and physical science—I mean that the science of chemistry should be taught to all as a portion of primary education; there is no easier means of bringing out the reasoning powers of a boy than by qualitative analysis—and that the highest branches of chemistry and physics should be thoroughly taught to those who intend following any chemical work. We want

young chemists so thoroughly illuminated that they carry a light with them everywhere and into everything, not, if I may use the simile, to "strike only on the box." Our next generation of workmen will, I hope, be an educated race, and we may be able to get foremen who are well up in chemistry. I am not at all afraid that the majority will ever be over-educated, and as for overwork, I fear that for the teachers much more than for the scholars. At present there are many branches of manufacturing chemistry almost entirely in the hands of the Germans and French, especially those where a thorough knowledge of organic chemistry is required. We have not the workmen and we have not the foremen in this country at present, neither the army nor the non-commissioned officers that can undertake such work. In an excellent paper read by our secretary, Dr. Dobbie, to the Glasgow Philosophical Society last week, he has collected statistics of the sources of the papers abstracted for the *Journal of the Chemical Society*. I would refer all interested in this subject to the paper itself, which, when published, will be widely read. He has allowed me to quote, however, one of the results. Here counts the total number of papers published in 1882 at 3,000. He compares the years 1872 and 1882, and shows that in the former year the abstracts of papers were of English 151, of German 809, which in the latter year increased to 232 of English and 1,442 of German. But of these the organic researches were in 1872, 31 of English, 67 of French, and 296 of German; in 1882 they were increased to 59 of English, 96 of French, and 574 of German. So that in this, the most difficult department of chemical knowledge, the Germans retain their power of creating ten times the number of papers that we do, and the French are also ahead of us; and these do not represent the actual total number of papers published abroad, but only those considered to be worth abstraction by English chemists connected with our Chemical Society. Dr. Dobbie points out that the papers show that we are specially behind in physiological and agricultural chemistry. This must be patent to all chemists. Researches in animal and vegetable chemistry are peculiarly difficult, and amidst all our voluminous manuals on theoretical and analytical chemistry we have scarcely a single book by any English author, except translations, on proximate organic analysis. There is a considerable amount of chemical work which is much required, and which, although it may be called research, is not original research, but is equally important, and this valuable work is especially that which, on account of its want of novelty, is not particularly attractive. I allude to the verification of methods, to exhaustive studies of combining equivalents, boiling points, vapour densities, etc. Although not the actual explorers in the dark mine of science, these workers prepare the tools, level the ways, and provide the lights—their work is absolutely necessary, and they should share the spoil of the victors. It is good work for the higher-class chemical students in the training colleges or universities of the future; and here I would mention that the delicate manipulations of quantitative analysis appear to me to add another to those numerous opportunities now being discovered for the application of the higher education of women.

One of the important subjects which will be brought before you this session is the question of commercial analysis. It is one which presents many difficulties. You are aware that Prof. Lunge, of Zurich, has addressed a letter to most of the European chemical societies with the view of securing an international agreement in analytical methods, in the statement of results, and in sampling. The subject is not at all new to us here. In 1874 we discussed it at the Chemical Section, and a resolution was then placed in my



hands asking the British Association to appoint a committee to inquire into the question. The Newcastle Chemical Society, now a section of the Society of Chemical Industry, held a meeting at Newcastle, at which Dr. Lunge was present, and adopted, on our request, a similar motion. The committee was appointed, in 1874, by the British Association, at Belfast, and consisted of Professor Dewar, Mr. Fletcher, with Mr. Allen as secretary, and myself as chairman. The two substances named in the report were potash salts and phosphoric acid. We addressed letters to all the best known analysts at home and abroad. The answers to some of these form amusing reading even now; but a good deal of valuable information was elicited, and all showed the great want of uniformity which prevailed, especially in phosphates. A few analysts refused to reveal their methods; one, at least, would not, as he said, "pour la gloire" alone. The researches on the phosphates were not fully completed—we found the field too extensive; but the report on potash was finished, and we fully proved that the methods of some of the German chemists must be 1 to 2 per cent. higher in the estimation of potash salts than they ought to be, especially where much soda salt was present. The analyses are fully detailed in our report. But what is the result? It was stated at the meeting of the London Section the other day, when this question was discussed, that the Stassfurt manufacturers have formed a syndicate, and determined to sell their potash salts only by an analytical method which we consider too high; and as our consumers here are equally determined not to buy by that analysis, they will allow for the difference in the price, or a broker will act as the medium between them, and buy by one and sell by the other analysis. The great question is whether the makers will follow the new analysis when the old one has been found to be wrong, if, as in the case of soda, for instance, it has been long the custom, or it pays better, to sell on a wrong method or a wrong atomic weight. If we cannot agree in this country on so simple a matter as the actual amount of soda in soda ash, which differs in different parts of the kingdom, what are we to do with the phosphates, for instance, whose names are legion, and what with the international weights and measures? Then, again, would it not be better to base the prices of all articles on 100 per cent. or purity? In many instances the price would then be an easy measure of the quality. The subject presents many lines of inquiry and discussion, and I do not wish to anticipate our special meeting called to consider it, particularly as it will be introduced by Mr. Tatlock, who was associated with it in the British Association Committee. But I would just point out that this is one of those questions in which, in this country at least, the Society of Chemical Industry, drawing its members from all classes of chemists, alone has any chance of doing any good. It is a very proper subject for our discussion, and I have no doubt that at any rate some improvement and advantage will result. To have chemists stigmatised as high and low, so that brokers prefer to sell by one and buy by the other, is a scandal to our exact science that we cannot too deeply deplore.

One general subject I would briefly refer to, although it affects other British manufactures as well as our own—that is, the heavy uneven charges and monstrous mismanagement of our railways. I refer to it here because the subject has been brought prominently forward by a once well-known member of this Society, Mr. Peter Spence, of Manchester, now gone to his rest. A pamphlet has been published containing his evidence given before the House of Commons Committee on Railway Rates. It is entitled "How

the Railway Companies are Crippling British Industry and Destroying the Canals." I would advise all to read it. The details given there of the unequal rates charged in different parts of the country, and the excessive nature of them all, are fully exposed. It is remarkable that it should be so much cheaper for us to send goods to India or America than to London, and that goods should be cheaper in one direction than in another. The American railways are made to open up the country for carrying goods to the ports; ours appear to have been made to throttle our inland commerce. Anything is possible on a railway, as in the case of the railway company that charged so much more carriage for the secondary battery after it had been charged with electricity to make up for the additional weight. The late Mr. Spence strongly protested against the railway companies increasing their monopoly by buying up canals, as some have done in our district, urging that it would be far better to enlarge and extend the canals through the country, a large steam lighter on a large canal being the cheapest known mode of conveyance. The "cold without" of the canal boat is cheaper to keep up than the "hot within" of the locomotive. I remember much interest being excited by an experiment shown by Mr. Spence, before the chemical section of the British Association, at Exeter, in 1869, in which he raised the temperature of caustic soda to 250° Fahr., by passing into it steam at 212° Fahr. Practical application has recently been given to this discovery in Honigmann's fireless caustic-soda boiler and engine.

The alizarine industry, one of the last triumphs of organic chemistry, is particularly interesting to this district, where we have so many Turkey-red dyes. The manufacture has been almost exclusively in the hands of German makers. A published statement of one of the works gives the net profits of 1882 at £360,700, after writing off to a sinking fund during three years the sum of £250,000. I may add that this work is remarkable for its very large and expensive scientific department, and the profits give us an idea how much we have been compelled to contribute to the support of that establishment. This manufacture has always been particularly urged against our home chemists, because most of the anthracene is exported from this country and then reimported as alizarine—that is to say, that in addition to the discovery the German chemists have manufactured the new product, and have shown us how to utilise one of our raw materials, and charged us pretty heavily for our want of chemical knowledge. It should not be forgotten that neither alizarine nor indigo was discovered by accident, but by patient investigation on well-known lines, and both result from a long series of separate discoveries by different experimenters. The amount made is now about 1,300 tons a month of 10 per cent. alizarine, of which 600 to 700 tons are consumed in the United Kingdom. It is some consolation to know that two firms in this country, Messrs. Hardman and Company, Manchester, and the British Alizarine Company, London, are now making a considerable portion of this home consumption, the latter now increasing from 200 to 300 tons per month. The total make is worth about £1,560,000, but this would require about four times that value of madder, which could not be produced. Artificial indigo is now also made in Germany, but here the yield of the plant is much larger than that of alizarine from madder, and the artificial product has more difficulties to contend with, but it is imported and used. Its introduction is only a question of time, and it may lead to further discoveries and better shades of colour. In 1883 the value of our imports of indigo, however, amounted to 100,713 cwt., valued at £2,467,083. We also imported of cinchona bark 117,325 cwt., valued at

£1,429,261, and of opium to the approximate value of £500,000. The value of these three imports amounts to £4,396,344. The total export of alkali amounted during the same time to 6,946,615 cwt., valued at £2,124,834. Thus the value of the imports of these three drugs amounts to more than double the value of the export of our largest chemical product, and the cinchona bark import for the year is £400,000 less than usual. This shows the great importance of these products, and yet it requires no prophetic gift to predict that all will be ultimately superseded. Indigo is already produced in the laboratory. Quinine and morphia are both gradually nearing measurable distance. Are these victories to be scored to us, or must we stand by and let the foreign students of organic chemistry snatch them from us? The replacement of these natural products, madder, indigo, cinchona bark, and opium, by artificial alkaloids produced in the laboratory, means the restoration of enormous tracts of land in different parts of the world devoted to the growth of these products, and in many instances freeing them for the cultivation of cereals and other foods. These are only two of the many prizes yet to be won. We also imported last year chemical manufactured products to the extent of £1,544,694. I have not the details of these at hand, but they represent a large number of finer chemicals that ought to be made at home. In therapeutics there is a very large field for the new organic products of the laboratory. Every day adds to the physician's armoury some new sharp weapon. Kairine or oxychinoline-methyl-hydride has been found an efficient substitute for quinine. Iodoform is now largely used as an antiseptic. Even the commoner metals are not yet worked out for medicines, for nickel in the form of bromide has just been introduced for the treatment of nervous diseases.

In quinine and morphia and chloroform some of our well-known manufacturers in London and Edinburgh hold their own against great odds. Some concession might be made to the manufacturers of these finer chemicals, especially chloroform and the alkaloids, by allowing the use of duty-free spirit, in which the German chemists have a great advantage. I cannot see any real objection to this, as the excise officer would have the entrée to the works. Methylated spirit is no use for some of these manufactures, and when men want to drink, it goes down the arid throat as easily and quickly as any other. Indeed, according to Dr. Richardson, methylic alcohol from its lower boiling point is more wholesome than ethylic; and "potable wood naphtha" was one of the chemical features of the London Exhibition of 1862, so that it is easily purified. An agitation is now going on in America for duty-free spirit for manufacturing purposes. The formation of worts for distilling and for brewing is becoming an important branch of applied chemistry, and there is none requiring more organic research. Cheaper starches, gums, and sugars will be sought to supply the alcohol, and we shall not always sacrifice so much valuable land in cultivating barley to make so little out of it. In 1882 there were 2,662,927 acres of barley under cultivation in the United Kingdom, besides an import valued at £5,511,498. Perhaps even some day we may have dry British wines with the pure delicate flavour of our home fruits, when some chemist has discovered a means of completely precipitating the acids which distinguish these fruits from the grape. So much has been recently added to our knowledge of fermentations, and the great economic and social importance of ferments, that it is impossible to predict the extent of the future boundaries of fermentation. Is it impossible to produce gutta serena and india rubber, of which natural products over £2,000,000 worth are annually

imported into Liverpool? I do not mean imitations, but condensed, partly-oxidised, hydrocarbons, as these bodies seem to be. How much longer are we to stick to the old-fashioned method of making cyanide? and when are we to obtain ammonia from the air, if indeed the ironmasters do not make it a drug in the market? Where is our cheap aluminium? We are constantly promised this valuable metal at the price of copper or less, but when are we to get it? The manufacture is now almost confined to one work in France. If it is the case that we are to have two separate pipes for gas, one for heating and one for lighting, we may see a vast impetus given to the use of what is likely to be the domestic fuel of the future, and we may also see the illuminating power so increased that it may light our rooms without so offensively heating them. The deposition of salts by refrigeration, in contradistinction to evaporation, has been very little studied in this country. The subject of refrigeration will be brought before you by our treasurer, Mr. Coleman, whose name is so identified with it. The use of dialysis also is little known here, although a good deal used in the sugar works of France. The vacuum pan, now of very general application, is another appliance little resorted to here in other than sugar works. Electricity also will come to our aid; its use in the precipitation of the more valuable metals seems to be foreshadowed. This may cause more attention to be paid to the solution of weak ores in acids, and their extraction in the wet way. In electric lighting the manufacture of carbons is becoming an important chemical industry, though not as yet to a large extent in this country. In our large alkali works we may look for considerable additions to our mechanical appliances. The revolving saltcake, carbonating, and black-ash furnaces, the filter press, and the hydro-extractor have already done much for us; but we are only on the threshold of the proper use of fuel, and we may ere long be using gas not only for fuel but for driving our engines instead of steam. That ammonia will be largely recovered is now certain, and perhaps it may become a low-priced product; and I would remark here that the ironworks are teaching our chemists to look after their chimney gases. No less than 50 European patents were taken out last year having ammonia for the subject. If cheap there will be many uses for the volatile alkali, independent of those for agriculture and the soda ammonia process, each large outlets. Our President has cleverly summed up the present position of the chlorine industry, and the prospects of competition between the old Leblanc process and its new, or rather its newly-revived, competitor, the soda ammonia process, which has now taken such firm root in this country. His prediction that bleach ought to be £10 per ton has been realised, and much more rapidly than he could have expected; and although other chlorine products have not yet risen in proportion, these are bound to follow. There is no doubt that hydrochloric acid must rise in value, from its increased cost of production, and as new applications are found for it. We must not, however, forget that 70 per cent. of all we make is run into the sewers, and lost as chloride of calcium; and in a similar waste the soda ammonia process is equally faulty. Whether or not chlorine will be produced from chloride of calcium remains to be seen, but if it be the advantage will still remain with the old process. We cannot as chemists, however, look with any complacency on a process as perfect which gives up less than a third of the chlorine available for use. Of course, it is always possible that another bleaching agent may be discovered which may replace chlorine. Peroxide of hydrogen is that which, of all others, is the least objectionable if it could be produced at a low price. Its cost at present,



however, renders it out of the question. The method proposed by Dr. Dobbie and Mr. Hutcheson of separating the chlorine on the cloth itself by electrolysis is a further step in the right direction if it can, as I hope it will, be practically introduced. Again, in the Leblanc process we throw away all the sulphur in the most offensive way. It is a very imperfect process which obliges us to cast away a waste product amounting to as much as the saltcake we use. That this has been done so many years is, to my mind, a proof that we have not scientifically worked out our methods. Schaffner's process, a foreign one, for recovering the sulphur and utilising the whole material, has been worked out and ably advocated by Mr. Chance, of Birmingham, and most of us are familiar with its details. The actual recovery of the sulphur in a saleable form does not appear to have been thoroughly perfected; but the process elicited general approval from a large meeting of alkali makers and technical chemists at Liverpool last year. Many things have happened since they and the pyrites companies have wisely reduced the price of sulphur. This concession may postpone for a time the erection of Schaffner or other plant for the recovery of sulphur. But do not let us suppose that the question is shelved. It seems to me that if the Leblanc process is to hold its own, this important question must be solved, and this waste nuisance done away with. Nothing should be more encouraged than the utilisation of waste. If the man is to be honoured who makes two blades of grass grow where one grew before, should we not give a double measure to him who grows grass where nothing grew before, and even greater honour to him who creates something valuable out of less than nothing, in fact out of a costly nuisance.

One of the desiderata of the day is the extraction of glycerine from the waste leys of the soapworks, and a good many patents have been lately taken out for this purpose. Glycerine has become a substance of great commercial importance, and it has become dear on account of the diminution in the composite candle industry. This is another instance of a valuable product which has always been thrown away in the soap leys to add to the varied contents of the sewers. This reminds me that if any chemist is very hard up for a subject, these sewers, with their chaotic contents, will give him ample opportunity for fine analytical skill, if he essays to pick out from the very cheap mixture a few of its valuable ingredients. A thoroughly scientific pick is required, and one has not yet been forged which will enable him to pick out any pay except "through the nose." If, however, a separate system of small water-tight and air-tight sewers be ever adopted, which will deal only with the domestic water supply and not with the rainfall, even sewage may be worth his attention. Here we have made some progress, this being the only British city, as far as I know, in which the public urinals are utilised for the production of ammonia. The chemists are somewhat in the position of the Yankee storyteller, who says, "If we don't know any facts we kin' make them." Our science has been making facts for the last 50 years, and who shall say where the next 50 years may land us? We must, however, welcome and make the most of every new addition to our knowledge, however apparently trivial. The philosopher has been described as the observer of minute differences. That awful sneer, "*Cui bono?*" which has deterred many a young discoverer, must be thrown away on us. "What is the use of it?" is the first question of ignorance. Our experience of the greatness of little things should teach us rather to ask "What may not be the use of a new fact, however trivial?" If Madame Galvani had not caught a cold,

and the doctor had not ordered her a bowl of frog soup, and if her husband had not hung the frog's legs on a copper hook to an iron rail, we might not have had the electric telegraph. A *cui bono* little thing may some day mean the greatest good to the largest number. The marvellous power of little things is well illustrated in those remarkable microcosms which appear to be the cause of so much human suffering, or perhaps as human lives are not always considered so important as animals or crops, I should refer to the bacteria of splenic fever, or to the tiny fungus of the potato disease, at whose mercy there lies a crop worth generally in this country about £20,000,000. These studies are only in their infancy, and we shall be largely called on to assist in fighting these small plagues. New remedies and new disinfectants will be required at our hands in the sweet by-and-by, when we are to enjoy immunity from all diseases. There is, however, no doubt a reward for all labour, however apparently unremunerative, and at any rate it is a good thing to have something to tussle with. We inherit an amount of chemical knowledge which necessarily divides us into many different departments. The tract bequeathed to us is so large that each has enough to do to look after the cultivation of his own particular field; but it is none the less advantageous that we should all meet together to discuss our various difficulties, and reveal our new discoveries. I augur for the Society of Chemical Industry that its meetings will so bring us together for mutual advice and mutual assistance, and if we do not always succeed in borrowing new tools or obtaining new lights from each other, to help us to lay the difficulties and dig up the underlying treasures, we shall at any rate return to our several fields of labour assisted and encouraged.

And now, in concluding this discursive talk, I would bid hearty welcome to members and strangers who have favoured us with their presence to-night. It is said, "You should never prophesy unless you know," but I do know my colleagues on the committee of this Section, and I can confidently predict that, whatever may be my own failings, they will make this Section a perfect success; and as the health and prosperity of the Section will depend much on the quality and variety of the food produced, I will only preface the intellectual feast which will be laid before you by the appropriate grace—may

"good digestion wait on appetite,  
And health on both."

A cordial vote of thanks to Mr. Stanford for his address was moved by Prof. DITTMAR, seconded by Mr. J. J. COLEMAN, supported by Prof. FERGUSON, and carried by acclamation.

## Journal and Patent Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*An Improved Boiler or Digester for Effecting Chemical or other Operations.* G. Knowles, London. Eng. Pat 3287, July 3, 1883.

THE boiler or digester which constitutes the present invention is designed for use in such operations as that of digesting raw fibrous materials for paper-making with a solution of sulphurous acid, or of a bisulphite, or with other solutions which attack iron. It is composed of a closed steam-tight vessel, containing another vessel of somewhat smaller diameter, the space between the two vessels being filled or nearly so with water or other liquid. If the inner vessel be filled to the same level as the outer vessel with a liquid or solution of the same

density, the hydrostatic pressure on the inside and outside of the inner vessel will naturally be equal. The upper part of the outer vessel forms a steam chest or dome, and the inner vessel is in free communication therewith, the edge of this vessel projecting upwards, above the surrounding liquid. The inner vessel is provided with one or more pipes or coils, by means of which its contents can be heated by steam or hot water."

*Improvements in Apparatus for the Collection and Condensation of Vapours Evolved in the Manufacture of Indiarubber Goods partly Applicable for Use in the Condensation of other Vapours.* C. A. Burghardt, Manchester. Eng. Pat. 3335, July 5, 1883.

THIS invention relates to apparatus for the condensation of the "naphtha, benzol, or other volatile solvent which is evolved in the form of vapour during the manufacture of Indiarubber goods." The apparatus "consists of a chamber which is divided into a number of compartments by means of partitions, each consisting of a number of sheets of wire gauze, or of thin finely-perforated plates. The said shell is made in sections, and the sheets of gauze are larger than the area of each section, so that the edges of the said sheets project all round or on all sides into the water or cooling fluid with which the condenser is surrounded."

## II.—FUEL, GAS, AND LIGHT.

*The Decomposition of Ammonia by Heat.* William Ramsay, Ph.D., and Sydney Young, D.Sc.

It is well known that when ammonia gas is passed through a red-hot porcelain tube, or when it is exposed to the electric spark for some time, decomposition into nitrogen and hydrogen ensues. Numerous attempts have also been made to bring about the combination of free hydrogen and nitrogen, but the results have not been very satisfactory. The subject has been studied especially by Deville (*Annales*, 135, 104; 127, 281; 135, 104); and more recently by Donkin (*Proc. Roy. Soc.* 21, 281), and E. Johnstone (*Chem. News*, Dec., 1883). It is also stated in Watt's Dictionary, vol. i., page 183, that ammonia is decomposed by passage through a red-hot porcelain tube containing copper or iron wire, and to a less extent when gold, silver, or platinum wire is employed. The object of the authors was to ascertain the temperature at which the decomposition of ammonia takes place, and to study more carefully than has yet been done the influence of the material of the vessel or tube containing the gas. The ammonia gas was generated by gently warming a concentrated aqueous solution of ammonia, and was dried by passing it through three bottles containing quicklime, and then led through a tube heated by a Fletcher's furnace. The undecomposed ammonia was estimated by absorption in a known quantity of standard sulphuric acid and subsequent titration of the unsaturated acid, while the mixed nitrogen and hydrogen were collected in a suitable vessel and their volume measured. The temperature of the furnace was ascertained approximately by the fusion of salts placed on small pieces of platinum foil lying on the surface of the tube. The results obtained were as follow: (1) With a porcelain tube filled with broken pieces of porcelain—

Temperature. Deg. C.	Mean Percentage of Ammonia decomposed.
500 .....	1.55
520 .....	2.53
600 .....	18.28
620 .....	25.58
680 .....	35.01
690 .....	47.71
810—830 .....	69.5

There was a considerable want of uniformity at the higher temperatures, owing, first, to the difficulty in keeping the temperature of the tube constant, and, secondly, to the variable rapidity of the current, the influence of which was very great, as might be expected, since there is

practically no recombination of the nitrogen and hydrogen to form ammonia. (2) With an iron tube filled with broken pieces of porcelain—

507—527 .....	1.15
600 (current very fast) .....	21.36
600 (current much slower) .....	31.14
628 .....	65.13
676—695 .....	66.37
730 .....	93.38
780 .....	100

(3) With a plain glass combustion tube, lying in the iron tube—

780 .....	0.21
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(4) With a glass combustion tube, filled with fragments of broken glass tubing—

780 .....	1.72
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(5) With a glass tube filled with strips of ignited asbestos cardboard—

520 .....	2.90
780 .....	100

(6) With a plain iron tube; (a) no oxide of iron present—

780 .....	100
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(b) Tube partially oxidised, and therefore water formed—

780 .....	95
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(7) With a glass tube containing several lengths of iron wire—

760 .....	75
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(8) With a glass tube containing several lengths of copper wire—

760 .....	2.0
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(9) With a glass tube filled with copper reduced from the oxide, to give a large surface—

780 .....	50.2
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The results may be summed up as follow: (1) Under the most favourable circumstances—with an iron or porcelain tube, or a glass tube containing asbestos—the temperature at which ammonia gas begins to decompose lies a little below 500°. (2) In contact with a glass surface, the temperature at which decomposition begins is much higher. (3) The influence of the rate of passage of the gas, or, in other words, of the time of exposure is very great. (4) The nature of the surface of the containing vessel exerts an enormous influence on the amount of decomposition. (5) As might be expected, the amount of decomposition is greater when the surface is increased. Attempts were made to study the influence of time and of pressure on the decomposition of the gas by heating it in closed glass tubes protected by copper wire, copper electrolytically deposited, and sheet copper, but the tubes almost invariably burst. One tube, however, was heated to 780° for six hours without any appreciable decomposition of the gas. It is to be observed that the decomposition of the ammonia was never absolutely complete, for the issuing gas had always a faint ammoniacal smell and an alkaline reaction sufficiently powerful to turn a piece of red litmus paper blue, and this was the case even when the ammonia was passed through an iron tube heated to very bright redness with the blast. This fact agrees with that observed by Deville that the decomposition of ammonia gas by means of the electric spark is never quite complete, the most probable explanation being that recombination of the nitrogen and hydrogen takes place to a very slight extent. When, however, a mixture of dry nitrogen and hydrogen was passed through a red-hot glass tube containing iron filings, or through an iron tube, the presence of ammonia could not be recognised. When the mixed gases were moist, a trace of ammonia was formed, but in this case nascent hydrogen may have been produced by the decomposition of the water by the iron. The authors hope to have an opportunity before long of studying the influence of gas carbon on the decomposition of ammonia, this substance being always present in gas retorts, and practically forming the walls of the retorts.—S. Y.



*Notes on Ammoniacal Liquor.* A Paper read before the North of England Gas Managers' Association, by J. H. Cox, jun.

AMMONIACAL liquor, or "gas liquor," is the watery solution of ammonium salts which results from the distillation of coal, and condenses in the hydraulic main and scrubbing and condensing plant. The ammonia is combined with the different acids, also formed during the process, to produce salts which may be classified as volatile and fixed. These are principally

#### VOLATILE SALTS.

Ammonium sulphide  $(\text{NH}_4)_2\text{S}$   
 Ammonium hydrosulphide  $(\text{NH}_4)\text{HS}$   
 Ammonium carbonate  $(\text{NH}_4)_2\text{CO}_3$   
 Ammonium bicarbonate  $(\text{NH}_4)\text{HCO}_3$   
 Ammonium thiosulphate  $(\text{NH}_4)_2\text{S}_2\text{O}_3$   
 Ammonium sulphite  $(\text{NH}_4)_2\text{SO}_3$

#### FIXED SALTS.

Ammonium sulphate  $(\text{NH}_4)_2\text{SO}_4$   
 Ammonium sulphocyanate  $\text{NH}_4\text{CNS}$   
 Ammonium cyanide  $\text{NH}_4\text{CN}$   
 Ammonium ferrocyanide  $(\text{NH}_4)_4\text{FeC}_6\text{N}_6$   
 Ammonium chloride  $\text{NH}_4\text{Cl}$

of which the sulphide, carbonate, chloride, and sulphocyanate are the most common. The first class of salts yield up their ammonia on simple boiling; the latter require the presence of lime to combine with the acid and set free the base. The proportion of fixed to volatile ammonia varies greatly (see Table I.) in different parts of the plant, the former being found chiefly in the hydraulic main, and the latter in the condensers. The quantity and kind of the ammonium salts depend on the kind of coal distilled, the temperature to which the liquor has been subjected, its exposure or non-exposure to the air, and the manner in which the washing and scrubbing are done. The greater the heat in the retorts the more cyanide and sulphocyanate are, as a rule, present in the liquor. Exposure of the liquor to the air occasions loss of ammonia from conversion of the sulphide and carbonate into hydrosulphide and bicarbonate respectively. Table I. shows how varied is the percentage of ammonia combined with each acid in different parts of the condensing plant. Gas liquor varies in appearance from bright yellow (due to the presence of polysulphides of ammonium) through all the shades of yellow to a colourless liquid. The oxidation of ammonium hydrosulphide often occasions a cloudiness from deposition of sulphur. The amount of ammonia present in the liquor is expressed by indicating the number of ounces of concentrated  $\text{H}_2\text{SO}_4$  required to neutralise the ammonia present per gallon. The strength of the liquor by Twaddell's hydrometer is often taken as the test, the number of degrees indicated being supposed to give, when doubled, the ounce strength of the liquor. The fallacy of this test is apparent when we consider that the more free ammonia is present the lower is the specific gravity of the liquor. The saturation test, or simple neutralisation of the liquor with sulphuric acid, is misleading, as it takes no account of the fixed ammonia. The only true valuation is that in which the ammonia is distilled with addition of lime and subsequently estimated. An important work done by the gas liquor is the partial purification of the gas from sulphuretted hydrogen and carbonic acid. On an average one ton of Newcastle coal yields 6½ lb. of ammonia, 15 lb. of sulphuretted hydrogen and 30 lb. of carbonic acid. These two last would require 38 lb. of ammonia to combine with, and as only about 5½ of the total 6½ lb. are available for this, it would appear that about sixth-sevenths of the impure gases are not eliminated. But by repeatedly bringing the liquor into contact with the gas the purification is carried further. Sulphuretted hydrogen is displaced by carbonic acid and remains in solution, while the gradual conversion of the sulphide and carbonate into hydrosulphide and bicarbonate allows the ammonia to remove a double portion of impurity. Table I. gives examples of the purifying work done by liquor. The removal of carbon disulphide from the gas, now for the most part

effected by the sulphide of lime in the purifiers, would probably be accomplished equally well by ammonium sulphide, but the use of the latter would be more expensive, as a certain amount of ammonia would be converted into the form of sulphocyanate, and this is practically useless in the sulphate house. Thus to remove 3,000 grains of sulphur per ton (=30 grs. per 100 cubic feet of gas) about 1,600 grains of ammonia would be used at a cost of somewhat over a penny. This penny would have to be added to the purifying account. The ammonia of gas liquor is generally converted into the more convenient form of sulphate, and here a considerable loss generally occurs. When a large percentage of the ammonia is "fixed" a great deal of it generally goes away in the waste liquor. Table II. (No. 11) gives the analysis of a sample of liquor of which 30 per cent. of the total ammonia was thus lost. No. 14, on the other hand, was particularly good for working, and the loss was only 0.8 per cent. Most samples of liquor lie between these two extremes. In practice it seems impossible, even by the use of a large excess of lime, to drive off all the fixed ammonia, but, nevertheless, there is no ground for the assertion of some that it does not pay to use lime at all. It is a good rule to add 5 lb. of lime per 10,000 grains of fixed ammonia. All this points to the necessity of rigidly analysing the liquor before and after it has been in the still, so as to regulate the supply of lime and the length of boiling. In the course of the discussion which followed the reading of the paper, Mr. Ridley, of Newcastle, gave it as his opinion that it did not pay to attempt to get the fixed ammonia from gas liquor, and Mr. Cox mentioned, further, that at Hendon purifying cost 3.9d. per ton of coal, viz., 2.5d. in lime and 1.4d. in labour, while sulphate cost £5 per ton to manufacture, and they make 20 lb. of sulphate per ton of coal.—A. R. D.

AN American correspondent of the *Journal of Gas-Lighting* sends an interesting communication on the subject of the appearance of naphthalene in a New Jersey gasworks. The appliances stand in the following order from the hydraulic main, condensers, washer, exhauster, purifier, station meter, and are all very large for the quantity of gas made, the connecting pipes being sufficient for five times the quantity. The gas enters the holders at a temperature of about 21° C. Two or three times in the course of the 10 years that the works have been in operation, the inlet of the gasholder has, in warm weather, been choked with a deposit of naphthalene. It was steamed out at first, but last summer appeared again and again. This was while carbonising coal alone, and no cannel was used, but as soon as from 3 to 10 per cent. of this latter was added, the inconvenience entirely ceased. It was thus proved then that the addition of cannel tends to prevent the formation of naphthalene.—A. R. D.

*Results obtained in the Manufacture of Sulphate of Ammonia and Sulphuric Acid.* A Paper read by Mr. J. Hepworth before the North of England Gas Managers' Association.

THE author, referring to the chances of gas as the light of the future, dwells upon the necessity of cheapening its production by an economical utilisation of the by-products. The following results are from his own experience. Having to deal with 1,750 tons of liquor of 5° Tw., for which he could not obtain more than 10s. per ton, he put up sulphate plant at a cost of £1,300, exclusive of buildings. He thinks, however, that a plant of equal efficiency could now be erected for £750. Last year's work shows the following particulars: Liquor per ton of coal=28.36 gallons of 5° Tw. Sulphate of ammonia produced per 100 tons of coal=1 ton 1 cwt. 0 gr. 13 lb. Cost of sulphate per ton (exclusive of liquor)=£6 15s. Amount realised for ammoniacal liquor per ton of coal, before introduction of sulphate manufacture, 1s. 1.15d. Amount realised for sulphate of ammonia per ton of coal, 2s. 4d. The liquor as run into the first still contained 2.54 per cent. of ammonia, when

run into the second 0.589 per cent., and when taken from this latter only .068 per cent., clearly proving the advantage of using the lime in the second still. With a view to utilising the waste gases evolved in the sulphate manufacture, a sulphuric acid plant was erected at a cost of £380 for chamber and condensers and £70 for the kiln. When sulphur was used acid of 150° Tw. was produced at a cost of £3 10s. per ton, against £3 15s. for acid purchased at a distance. Now by the use of spent oxide—estimating the cost at 7d. per unit—the same acid is obtained for £2 per ton. The gain effected by this represents about 1½d. per ton of coal carbonised.—A. R. D.

*Contributions to the Chemistry of Mineral Fuels.* Boassingault. Dingl. Polyt. Journ. 331, 250, 1884. Taken from Comptes Rendus 1883, 96, 1452.

THE author has examined the bitumen from the so-called burning springs of Ho-Tsing, in the provinces of Szu-Tchuan, China, where, in a region having an area of 50 square miles, several thousand springs exist, whence proceed inflammable gases at a high velocity, accompanied by bitumen and brine. At ordinary temperatures the bitumen is in the fluid condition, but when cooled, naphthalene separates out. The solid and liquid constituents of the bitumen separated by cooling and filtration have the following composition:—

	C.	H.	O.	N.
Solid.....	82.85	13.06	1.06	0
Liquid.....	86.82	13.16	0	0.02

Samples of asphaltum respectively from Egypt (I.) and from the Dead Sea (II.) possessed the following composition:—

	I.	II.
Carbon.....	85.29	77.84
Hydrogen.....	8.24	8.93
Oxygen.....	6.22	10.53
Nitrogen.....	0.25	1.70
	100.00	100.00

An amber-like fossil resin from the alluvial regions of New Granada, found in a lump weighing about 12 kilos, at the gold-washing establishment of Giron near Bucaramanga (I.), and a fossil resin from the auriferous alluvium of the province of Antioquia (II.), gave the following results upon analysis:—

	I.	II.
Carbon.....	82.7	71.89
Hydrogen.....	10.8	6.51
Oxygen.....	6.5	21.57
Nitrogen.....	0	0.03
	100.0	100.00

Pit coal from Canoas, situated in the Cordillera range, on the plateau of Bogata, 2,800 metres above the level of the sea (I.), and coal from the province of Antioquia (II.), yielded the following analytical results:—

	I.	II.
Carbon.....	80.96	87.05
Hydrogen.....	5.13	5.00
Oxygen.....	12.50	6.56
Nitrogen.....	1.41	1.39
	100.00	100.00

Two samples of the so-called "fusain," from the mines respectively of Blanzi (I.) and Montrambert (II.), Loire, contained—

	I.	II.
Carbon.....	87.81	93.05
Hydrogen.....	3.88	3.35
Oxygen.....	7.67	3.13
Nitrogen.....	0.61	0.17
	100.00	100.00

Anthracite from Chili (I.), and from Muso, New Granada (II.)—

	I.	II.
Carbon.....	92.25	94.83
Hydrogen.....	2.27	1.27
Oxygen.....	4.94	3.16
Nitrogen.....	0.51	0.71
	100.00	100.00

A sample of graphite from Karsob, which yielded pure

carbon by subjecting it at a red heat to the action of chlorine contained—

Carbon.....	97.87
Hydrogen.....	0.37
Oxygen.....	1.70
Nitrogen.....	0.06
	100.00

W. D. B.

*Improved Means of Obtaining Salts of Ammonia from Combustible Gases.* John Addie, Glasgow. Partly communicated by James Addie, Bilbao, Spain. Eng. Pat. 3246, June 30, 1883.

BY this invention the ammonia "which is contained in combustible gases, such as the gases obtained from blast furnaces, coking ovens, and gas producers, is directly converted and recovered as sulphate of ammonia by a continuous process, consisting of the following stages, viz.: (1) The combustible gases are charged or intermixed with sulphurous acid gas in sufficient quantity to combine with and fix the ammonia contained in them. (2) The gases are submitted to the action of a scrubber, or to equivalent action, to dissolve the sulphite formed. (3) The solution of sulphite of ammonia is oxidised by forcing or injecting air thereinto."

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*On the Pyridine Bases in Coal Tar.* Heinrich Goldschmidt and E. J. Constam. Berl. Ber 16, 2976.

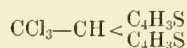
THE existence of two isomeric picolines having been predicted by E. Jacobsen and C. L. Reimer (Berl. Ber. 16, 2604), who obtained two different products by treating the picoline prepared from coal tar with phthalic anhydride and zinc chloride, the authors give a preliminary account of their investigation of the isomeric pyridine bases in coal tar. A considerable quantity of sulphuric acid, which had been almost saturated with bases in the purification of tar, was treated with steam to remove hydrocarbons, supersaturated with caustic soda, and the bases distilled with steam. The distillate was acidified with hydrochloric acid and evaporated, but the smeary impurities could not be removed by filtration. The liberation of the bases and distillation with steam were therefore repeated, and the acidified distillate was concentrated. After filtration the clear liquid was treated with caustic soda, and the liberated bases dried with solid caustic potash. 1 kilo. of the bases was obtained from 10 litres of the raw material. The product boiled between 92° and 200°. After repeated fractionation it was found that a little of the substance came over below 100°, about one half of the whole quantity between 114° and 117° (pyridine), while above this temperature no constant boiling point was observed. Very little came over above 160°. The portion below 100° boiled constantly at 92° to 93°, and was found to be a compound of pyridine and water. The addition of solid potash caused the separation of pyridine, boiling at 114° to 115°. The platinum double salt was prepared and analysed. The hydrate of pyridine, which has the composition  $C_5H_5N + 3H_2O$ , was prepared from pyridine by the addition of one-third of its volume of water and subsequent distillation. Two fractions were obtained, one at 92° to 93°, the other at 114° to 115°. The specific gravity of the hydrate is higher than that of a simple mixture of pyridine and water. The vapour density determination showed that the hydrate is decomposed by its conversion into vapour. The authors observe that the base obtained by Fritzsche from coal tar, and which boiled at 95°, was probably not identical with the cespitin, ( $C_5H_{13}N$ ), discovered by Church and Owen in the distillation products of turf, but was probably the hydrate of pyridine. The portion of the mixture of bases which came over between 114° and 117° was found to be pyridine. The fractions between 130° and 140° were mixed, but the quantity was not sufficient to allow of the removal of the lutidine. The bases were oxidised, according to Weidel's method,



with potassium permanganate, and the concentrated aqueous solution of the potassium salt treated with copper acetate. After removal of the copper from the precipitated salt by means of sulphuretted hydrogen and decolorisation of the filtrate with animal charcoal, the solution on evaporation yielded colourless needles of picolinic acid, melting at 137°. The filtrate from the copper picolinate was evaporated, acidified with acetic acid, and treated at its boiling point with copper acetate, whereby a blue-green precipitate was obtained, which, after being boiled repeatedly with water, was treated with sulphuretted hydrogen. The filtrate from the copper sulphide yielded on evaporation colourless crusts, melting at 310°. They were found on analysis to consist of pyridine carbonic acid. The properties of the acid show that it was isonicotinic acid ( $\gamma$ -pyridine carbonic acid) and therefore either  $\gamma$ -picoline, which has not yet been prepared, or lutidine must have been present among the bases. The quantity of picolinic acid was far greater than that of isonicotinic acid. These results show that orthomethylpyridine is the chief constituent of the mixture of picolines obtained from coal tar.—S. Y.

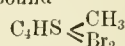
*On the Thiophene and Pyrrol Groups.* Victor Meyer. Berl. Ber. 16, 2968.

THE great similarity exhibited by thiophene, and the compounds already prepared from it, to benzene and its derivatives, led the author to the conclusion that thiophene differs from benzene only in the substitution of one of the acetylene groups forming the benzene ring by an atom of sulphur. This conclusion has so far been verified. Thiophene sulphonic acid ( $C_4H_3S-SO_3H$ ) and thiophenic acid ( $C_4H_3S.COOH$ ) prepared by the author and Herr Kreis, resemble benzene sulphonic acid and benzoic acid so closely that they are hardly to be distinguished except by analysis, or by the isatin test described below. The following are some of the results obtained by the author and his students—*Simple Condensation Products of Thiophene*: Thiophene forms condensation products like the aromatic hydrocarbons, in the manner described by Baeyer, and by Friedel and Crafts. Thus, with chloral in presence of glacial acetic acid and sulphuric acid, the substance—

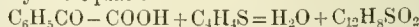


is formed, and it is decomposed by alcoholic potash in the same way as the benzene derivative. Bromal gives a similar product. The resemblance of dithienyl methane prepared from methylal to diphenyl methane extends even to its characteristic odour of oranges. It is, however, easily distinguished by its higher melting point and by its reaction with isatin. This substance, in presence of sulphuric acid, has no action on dithienyl methane in the cold; but on heating, the beautiful indophenine reaction is at once observed. The reaction is characteristic of almost all the derivatives of thiophene—thus thiophene sulphonic acid and thiophenic acid remain unaltered in the cold, but produce the deep blue colouration when warmed with isatin and sulphuric acid. Since the term thiophenyl is already employed for phenyl compounds containing sulphur, the author proposes the name thiänyl for the radical  $C_4H_3S$ , which stands to thiophene in the same relation as phenyl to benzene. Care must be taken in testing thiophene according to Friedel and Crafts' reaction, not to employ an excess of aluminium chloride, or the thiophene may be decomposed with liberation of sulphuretted hydrogen. Good results are obtained with benzyl chloride, benzoyl chloride, and phthalyl chloride. Benzoyl chloride, treated with thiophene and a small quantity of aluminium chloride, yields phenylthiänylketone ( $C_6H_5.CO.C_4H_3S$ ), analogous to benzophenone. The ketone gives with hydroxylamine the isonitroso compound  $C_6H_5.C(NO)H.C_4H_3S$ , and this, when heated with soda-lime, is decomposed with formation of benzoic acid and thiophene. The ketone gives a blue coloration when heated with isatin and sulphuric acid. *Homologues of Thiophene*: It is difficult to prepare monobromothiophene, for by the bromination of thiophene the dibromo compound is formed in larger

quantity, and hence the preparation of the next homologue of thiophene has not been carried out by means of this compound. But the author was led to believe that methylthiophene was present in the toluene obtained from tar; and at his request Dr. Caro, of Ludwigshafen, converted the sulphuric acid extract from 2,000 kilos. of toluene into the sulphonic acid, and then eliminated the sulphonic group by distilling the lead salt with sal-ammoniac. By this means an oil was obtained which, after rectification, boiled constantly at 110°. It contained 5 per cent. of sulphur, and appeared to consist of 15 per cent. of methylthiophene, and 85 per cent. of toluene. The product, when tested by Laubenheimer's reaction, gave far more characteristic results than ordinary toluene. Methylthiophene itself has not been isolated, but the dibromo compound

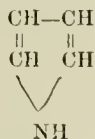


has been prepared in a state of purity. It boils without decomposition at 227° to 229°. The xylene and cumene obtained from coal tar also exhibit Laubenheimer's reaction, and therefore probably contain the corresponding homologues of thiophene. *Thiophene Sulphonic Acid*: A number of salts have been prepared, and also the chloride, amide, anilide, and the ethyl ether, and the sulphonic acid  $C_4H_3S.SO_3H$ , and mercaptan  $C_4H_3S-SH$  are in course of preparation. The phenol  $C_4H_3S-OH$  has not been obtained. *Colouring Matters from Thiophene*—(a) *Thiophene and Phenylglyoxylic Acid*: The formation of the violet-red colouring matters described by Claisen, by the action of phenylglyoxylic acid and its derivatives on benzene, is due to the presence of thiophene in that substance. Solutions of phenylglyoxylic acid and thiophene in glacial acetic acid were mixed and treated cautiously with concentrated sulphuric acid, and the product was poured after some hours into water, when a beautiful red flocculent precipitate was formed, which, after being washed and dried, was found, on analysis, to possess the composition  $C_{12}H_8SO_2$ . Its formation is probably represented by the equation—

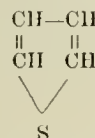


It dissolves in chloroform with a carmine, and in sulphuric acid with a deep violet-red colour. The colour of the latter solution changes suddenly on warming into a pure blue. (b) *Phenanthrene Quinone and Methyl Thiophene*: A mixture of the acetic acid solutions of phenanthrene quinone and the crude methyl thiophene obtained from toluene, yields a colouring matter which dissolves in alcohol and ether with a deep violet colour. The determination of the sulphur in the product agrees with the formula  $C_{19}H_{12}SO$ . When distilled with zinc dust a crystalline hydrocarbon is formed. Hence thiophene may be found useful in the production of the higher hydrocarbons. (c) *Phenanthrene Quinone and Thiophene*: Laubenheimer, who described the characteristic colour reactions of xylene and toluene with phenanthrene quinone, failed to observe the corresponding reaction with benzene, owing to the insolubility of the colouring matter in ether. It dissolves, however, in chloroform, forming an emerald-green solution. The colouring matter was prepared in quantity by the action of phenanthrene quinone on thiophene. Many other ketones give colouring matters with thiophene. According to the author those ketones which contain only carbon, hydrogen, and oxygen form colouring matters with thiophene when they contain the group  $CO-CO$ . On the other hand, simple ketones, and double ones, in which the carbon atoms of the two carboxyl groups are not directly combined, give no colouring matters. The intensity of the colour depends obviously on the nature of the molecule containing the group  $CO-CO$ . If it belongs to the aromatic or uric acid groups the colour is deep and beautiful, if to the fatty group the colour is weak and dull. Thus, pyruvic acid and thiophene give a dull violet colouring matter. Many aldehydes also yield colouring matters with thiophene, but they appear to have a more complex constitution than those formed from the double ketones. *Nitrogenous Colouring Matters from Tar*: The author obtained a tar oil from Dr. Häussermann, in Griesheim a. M.,

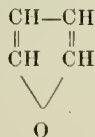
which boiled between  $90^{\circ}$  and  $109^{\circ}$ , and consisted chiefly of benzene and toluene, together with hydrocarbons of the fatty group and considerable quantities of pyridine bases which could readily be extracted by dilute acids. When this oil was treated with an aqueous solution of isatin and dilute sulphuric acid, a deep blue precipitate resembling indigo was formed. After treatment with a dilute acid, however, the oil gave no such precipitate, nor did the bases liberated from the acid extract. It was suggested by Dr. Caro that the reaction was caused by the presence of pyrrol in the oil, and this was found to be the case. When pyrrol is treated with isatin and dilute sulphuric acid the same colouring matter is formed, the yield amounting to 120 per cent. of the isatin employed. The substance is evidently formed by the combination of isatin and pyrrol, with elimination of water. The analogy of pyrrol, for which the author had already suggested the constitution—



with thiophene—



is shown by the fact that colouring matters are formed by the action on pyrrol, in presence of dilute acids, of the same substances which give colouring matters with thiophene. With the exception, however, of the isatin compound, the pyrrol colouring matters are unstable, and in this respect resemble the colouring matters of flowers. The analogy of furfuran—



with thiophene has been pointed out previously by the author. It thus appears that of the three acetylene groups forming the benzene ring one may be replaced by S, O or NH, without destroying the characteristic properties of benzene.—S. Y.

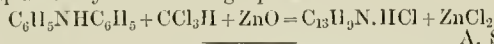
#### On Acridine. C. Graebe. Ber. 16, 2828.

THE interesting researches of Bernthsen and Bender (this Journal III., 1, 24) having shown that the composition of acridine is represented by the formula  $\text{C}_{13}\text{H}_9\text{N}$ , and not  $\text{C}_{12}\text{H}_7\text{N}$ , the author has again investigated this subject, as the values which were obtained by Caro and Graebe in their former researches on acridine extracted from coal-tar have agreed more closely with the formula  $\text{C}_{12}\text{H}_7\text{N}$ . The author found that the composition of crystallised acridine when fused is represented by the formula  $\text{C}_{12}\text{H}_7\text{N}$ , whilst acridine dried at  $100^{\circ}$  gave numbers agreeing with the formula  $\text{C}_{13}\text{H}_9\text{N}$ . This difference he attributes to the fact that acridine, especially during its fusion, absorbs small quantities of oxygen from the atmosphere, so that on analysis low results are obtained as regards the carbon. This difference being most noticeable in the case of the platinum-chloride, the author has carefully prepared this salt in a pure form. Analysis gave numbers agreeing more satisfactorily with the formula  $\text{C}_{13}\text{H}_9\text{N}$ . These results with the facts already established by Riedel, and Bernthsen, and Bender, leave no doubt as to the composition of acridine. The author has again prepared acridine sulphate, and studied its properties. It forms an insoluble salt, which can be dried at  $100^{\circ}$  without decomposition. However, on continued boiling with water sulphurous anhydride and acridine are

volatilised. Acridine-octohydride ( $\text{C}_{13}\text{H}_{17}\text{N}$ ) is prepared by mixing 5grms. of acridine, or 6grms. of the hydrochloride with 2grms. of amorphous phosphorus and 6grms. to 7grms. hydriodic acid (boiling point  $127^{\circ}$ ), and heating the mixture at  $220^{\circ}$  to  $230^{\circ}$  for 6 or 7 hours. The base crystallises from alcohol in colourless laminae, or long plates. It does not yield coloured or fluorescent solutions. Melting point,  $84^{\circ}$ ; boiling point,  $320^{\circ}$ . The hydrochloride crystallises in colourless plates, which are readily soluble in hot water. On heating the base with acetic anhydride and benzoyl-chloride the acetyl and benzoyl derivatives are produced. Methyl iodide forms methyl acridine octohydride.—D. B.

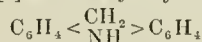
#### New Method of the Formation of Acridine. O. Fischer and G. Koerner. Berl. Ber. 17, 101.

IN attempting to prepare triphenylparaceneaniline by the action of chloroform on diphenylamine, the authors obtained instead considerable quantities of acridine. One part of chloroform, one part of diphenylamine, one part of zinc chloride, and about half a part of zinc oxide are heated to  $200^{\circ}$  or  $210^{\circ}$ , under pressure, for seven or eight hours. The contents of the tube are boiled with concentrated hydrochloric acid, the filtered solution poured into water, again filtered, and the acridine precipitated by an alkali, after which it is recrystallised from hot water. This process, which gives a better yield than that with formic acid and diphenylamine, recently described by Bernthsen and Bender (*Berl. Ber.* 16, 1802), especially if aluminium chloride be substituted for zinc chloride, is explained by the following equation:—



A. S.

*Erratum:* III. [1] 24 January. Hydroacridine should be



#### On the Examination of Petroleum. Dingl. Polyt. Jour. 250, [4], 169-172.

ACCORDING to F. Beilstein, the forms of apparatus recommended by Abel, Engler and others, for testing the flashing point of petroleum, with due care give good results, but it is necessary that the construction of the apparatus should be always the same, or different experiments will show different values. Two of Engler's testers, apparently of the same capacity, gave a difference of  $2^{\circ}$  for one and the same oil, because the marks to which the sample was filled were not of the same height, and therefore left a different air layer above the oil. According to Liebermann and Stoddard the results depend upon the size and kind of flame employed for igniting the vapour. Should the length of the apparatus be from five to seven times its breadth, the results are concordant, but if this proportion be exceeded, higher results will be obtained. If the apparatus have the right height, it is a matter of indifference whether it be filled one-quarter or two-thirds full of oil, but the quantity of air passed must not be too small. The length of time the ignition flame is held to the opening of the apparatus is also important. Beilstein recommends an apparatus consisting of a glass cylinder 35m.m. in diameter and 175m.m. in length, with two marks, one of which is 60m.m. from the bottom, the other 70m.m. The oil is poured in up to the first mark. Air is conducted from a gasometer by means of an indiarubber tube attached to a fine glass tube, terminating in a rose perforated with fine holes. The bulb of the thermometer is arranged about the middle of the oil. The apparatus is put into a water-bath, of which the temperature is increased one degree every two to three minutes. Each time the temperature of the oil shows a further rise of one degree, air is passed through for five seconds, and so quickly that a froth is raised in the upper part of the apparatus, while at the same time a flame is held at the mouth, and the temperature at which the vapour ignites is noted. The first result is only approximate. The oil is replaced by a fresh quantity, and the observations commenced at about that temperature which was found to be the flashing-point in the preceding trial. In this way observations can be made



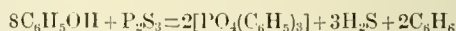
to 5° C. This apparatus gives a flash-point about five degrees higher than that of Engler. If the oil is so heated that the temperature of the water-bath is one degree higher than that of the oil, single observations vary about 25° C. Other forms of apparatus have not hitherto allowed of this degree of accuracy. In the preceding method there is ascertained the temperature at which the oil is dangerous for burning, but nothing is learnt about its capability of burning. For the latter purpose the distillation of the oil is requisite. The distillation is conducted in the following way: 200 grammes of the sample are placed in a weighed retort provided with a Glinzky's dephlegmator, to which is attached a tube about 0.75m. long without any condenser. The distillation is so regulated that about 2 grammes of the distillate shall pass over into the weighed receiver every minute. The distillates below 150° C. and between 150° C. and 270° C. are collected separately and weighed. The distillation is stopped at 270° C., the retort reweighed, and the amount of residue thus obtained. American petroleum gives 5 per cent. of a light oil boiling below 150° C. and less than 15 per cent. of a heavy oil boiling above 270° C. Such petroleum burns excellently in all kinds of lamps. According to Beilstein, petroleum which contains not more than 5 per cent. of light oil is quite free from danger. Mendeleeff states that fractions of petroleum from Baku, boiling between 15° and 150° have a density not always corresponding to the rise of temperature. The fraction boiling between 29° and 30° has a specific gravity of .626; that at 50° of .675; at 62° of .672; at 80° of .7483; at 90° of .7337; at 100° of .7659; at 140° of .7807; and at 150° of .7908. The coefficients of variations in specific gravity are in like manner related to the temperature. For the fraction boiling at 80° it is -.00093; at 86° -.00086; at 90° -.00084; at 98° -.00086; at 100° -.00088; at 110° -.00081; and at 114° -.00079. At higher temperatures these fractions gradually become less. According to Mendeleeff, petroleum from Baku contains the same kind of hydrocarbons as the American oil, but the low boiling fractions contain a small quantity of a hydrocarbon boiling at 55°, which has a higher density than hexane. The bulk of the hydrocarbons of the oil from Baku corresponds to the formula  $C_nH_{2n}$ . The behaviour of these hydrocarbons with bromine, nitric acid, potassium permanganate, and mercuric chloride, makes it probable that hydrocarbons of the acetylene series are also present. For the detection of aromatic hydrocarbons in petroleum products, G. Gustavson adds 10 to 15 grms. of aluminium to 1 or 2 grms. of bromine, and to the aluminium bromide thus formed 1 or 2 c.c. of the sample. The mixture is poured upon a watch-glass, and cautiously evaporated, when the needle-shaped crystals remain if the sample contained benzene or toluene. Alex. Ehrenberg, of Dresden, has constructed an apparatus for testing the flashing points of oils. It consists of two tubes joined together, a piston, a thermometer and small tube leading to the ignition flame. The apparatus is filled with the oil to be tested, and the temperature regulated by means of a water-bath. At intervals the piston is pushed down, and the gas given off by the oil is thus brought into contact with the flame. Upon raising the piston the observation can be repeated until the flashing point is found. The apparatus can be made of either glass or metal.—A. H. A.

#### Action of Phosphorus Trisulphide on Phenol and Cresol.

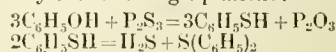
A. Geuther. *Annalen* 221, 55.

KEKULÉ and Szuch have shown that by the action of phosphorus pentasulphide on phenol, besides phenyl sulphhydrate, phenyl sulphide, and a high boiling product, a small amount of benzene is formed. As the formation of benzene from phenol depends on a reduction process, which, if brought about by pure phosphorus pentasulphide, could not be easily explained, it was thought that the pentasulphide must have contained some trisulphide, and that the reduction was due to the presence of this substance. On investigation this was found to be the case. 60 grms. of finely-pulverised phosphorus trisulphide and 10 grms. of phenol were heated for

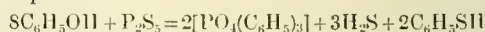
three to four hours in a retort provided with a reflux apparatus. The volatile products were then expelled by heat. A small quantity of undecomposed solid phosphorus trisulphide being carried over mechanically was removed by filtration. The filtrate on fractional distillation was divided into two parts, the first distilling under 100° and the second boiling over 360°. Phenylsulphide, which boils at 250°, and phenylsulphydrate, boiling at 165°, were present only in small quantities. The weight of the portion boiling under 100° was 16 grms. It was rectified, when it showed a constant boiling point of 81°. It was found to resemble benzene in properties and chemical constitution. The fraction distilling above 360° contained only traces of sulphur. Its solution in nitric acid liberated phosphoric anhydride very freely, a reaction indicative of the presence of phenylethylphosphoric acid. By taking into consideration the fact that almost the whole of the sulphur escapes in the form of sulphuretted hydrogen, and that the resulting benzene corresponds to one-fourth the phenol used, it may be assumed that the equation which illustrates this reaction is as follows:—



The formation of phenylsulphide and phenylsulphydrate is explained by the following equations:—



whilst the change which takes place when phosphorus pentasulphide is used would probably be illustrated by the equation



The reduction of phenol to benzene by means of phosphorus-trisulphide having proved a success, it was thought that this sulphide would act in an identical manner on the homologues of phenol. On subjecting cresol to the action of phosphorus-trisulphide, and proceeding in the above-described manner, it was possible to obtain toluene having a boiling point of 110°. Besides the fraction distilling above 360° and probably consisting of toluylethylphosphoric acid, an intermediate product boiling at 185° to 195° was obtained, which appeared to be a mixture of toluylsulphydrate and undecomposed cresol. Toluylsulphide was not formed. These results show that it is possible to abstract oxygen from phenols by the aid of phosphorus-trisulphide and convert them into the corresponding hydrocarbons.—D. B.

#### Products of the Destructive Distillation of Wood at Low Temperatures. C. F. Mabery. *Amer. Chem. Jour.* 5, 256.

THE author has examined fractions of crude wood spirit, (a) being taken at the commencement of the distillation; (b) after several hundred gallons had distilled over; and (c) when the distillation of methyl-alcohol had made considerable progress. (a) showed a high percentage of ethylaldehyde; (a) and (b) methylacetate; and (c) methylalcohol. Dimethylacetate and small quantities of acetone were also present.—D. B.

#### Picamar. By G. Niederist. *Monatsh. Chem.* 4, 487.

THE author came into possession of an original specimen of picamar prepared by Reichenbach himself, which was employed in the experiments described in this paper. The main result of the investigation was to establish the identity of this compound with the dimethylpropylpyrogallate obtained by Hofmann from beechwood tar, (*Ber.* 8, 66, and 11, 329). The statements of Reichenbach in regard to the properties of the substance are fully confirmed by the author. The following compounds are also described: Potassium picamar,  $C_{11}H_{15}O_5K$ . This was prepared according to Reichenbach, and obtained in the form of pearly plates. Acetyl picamar,  $C_{11}H_{15}(C_2H_3O)_3$ , by heating picamar for several hours with acetic anhydride in excess. It crystallises from alcohol in shining prisms (melting point 86-87°). Dibromacetyl picamar,  $C_{11}H_{13}Br_2(C_2H_3O)_3$ , crystallises from alcohol in colourless shining plates.—C. F. C.

*Improvements in Processes for Bleaching Ozokerit and other Solid Hydrocarbons, and for Rendering them Available as Substitutes for Wac.* J. Inray, London. Communicated by J. C. O. Chemin, Paris. Eng. Pat. 3435, July 12, 1883.

"CRUDE ozokerit is first melted in water kept at a temperature of 70° C., and the earthy and other solid impurities being allowed to settle, the melted matter is decanted, and solidifies in a condition fit to be treated by the following operations. First it is melted by superheated steam, and from 5 to 15 per cent. of flowers of sulphur being added, the material is distilled. The distillation, which is not fractional, gives a product, yellow, homogeneous, without fluorescence or dichroism. This being slowly cooled crystallises, the product being from 92 to 95 per cent. of the total quantity treated. The second operation may be conducted in either of two ways, either by subjecting cakes of the distillate to pressure, the plates of the press being kept at a temperature of 35° to 50° C., thus expressing the oils and hydrocarbons fusible at low temperatures, or by reducing the distillate to powder, and subjecting it to a shower of water at a temperature of 45° to 60° C., so as to wash away the oils and fusible hydrocarbons. Instead of water, amyl alcohol or other solvent of hydrocarbon oils may be employed at ordinary temperatures. The third operation consists in melting the product of the second at 60° or 70° C., and mixing intimately with it about 20 per cent. of amyl alcohol, then casting the whole into moulds. The cakes, after being subjected to pressure, are melted and digested for about four hours in agitation with animal charcoal, and the liquid is filtered through animal charcoal. On cooling, the product, amounting to about 80 per cent. of the matter treated, is white, hard, and sonorous. The liquids resulting from the third operation, or from the washing by alcoholic solvents, are distilled, so as to recover the alcohol and solvents, and the residue is mixed with the crude material to be subsequently treated. From 20 to 30 parts of residue from treatment of petroleum and naphtha may be mixed with 70 to 80 parts of crude ozokerit. A similar treatment is applicable to solid hydrocarbons generally, such as residues from petroleum, naphtha, pitch and tar, schist oils, and the like."

*Improvements in or Relating to the Treatment of the Products of Combustion or Destructive Distillation of Coal or other Fuel containing Nitrogen for the Purpose of Utilising the Ammonia contained therein.* J. H. Darby, Brymbo. Eng. Pat. 3084, June 21, 1883. Void, final specification not filed.

By this invention furnaces or other gases containing ammonia are to be passed through "any ordinary coke tower or scrubber," into the top of which is fed "water-spray or weak liquor" steam, "sulphurous anhydride, and gaseous oxides of nitrogen" being introduced into the flue conveying the gases to the foot of such tower, at "a sufficient distance from the tower to enable the gases to mix before entering" it. The liquid running from the bottom of the tower is thus to be solution of ammonium sulphate, which solution may be pumped to the top of the tower and passed through it a sufficient number of times to render it at last highly concentrated.

#### IV.—COLOURING MATTERS AND DYES.

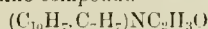
*The Indulins.* Otto N. Witt. Berl. Ber. 17, 74.

THE author describes a new method of preparing the indulins. When carried out in the usual way from amidoazobenzol and aniline, the preparation of the indulins is attended with evolution of ammonia, and the formation of complicated by-products. If, however, the phenyl-amidoazobenzol, previously described by the author, be heated with aniline hydrochloride, no trace of ammonia or ammonium salts is formed, and it appears also that the formation of complicated by-products is avoided. This method of preparation is more rapid and easy than the older one. The formation of the substance is explained

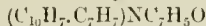
as being due to a breaking-up and subsequent reduction of the azo group by the hydrogen of the aniline, which, in its turn, unites with the amidodiphenylamine (phenylparaphenylenediamine) formed by the reduction of the azo body, thus giving indulin. From this, an analogy with the colouring matters of the tolylene blue series and the safranin group is apparent, these, like the indulins, being obtained by the abstraction of hydrogen from a mixture of a monamine and a paradiamine. The higher homologues behave in a similar manner to phenylamidoazobenzol. The product from diazoparatoluid and diaphenylamine forms yellow plates, melting at 109° to 110°, and soluble with a green colour in concentrated sulphuric acid. The corresponding derivative of metaxylinid (1, 2, 4) forms bright golden scales which melt at 142° to 143° and dissolve in concentrated sulphuric acid with a yellow colour. If in these compounds diphenylamine be replaced by other secondary aromatic amines, amidoazo bodies are obtained, which, without any difficulty, yield whole series of new colouring matters resembling indulin, when treated with the salts of primary bases.—A. S.

*On some Derivatives of  $\alpha$ - and  $\beta$ -Naphthol.* Emil Friedländer. Berl. Ber. 16, 2075.

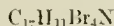
By heating the naphthols with aniline or ortho- or para-toluidine in presence of calcium chloride to a temperature of 230° to 250°, secondary amines are formed in abundance. As a rule, for one molecule  $\alpha$ - or  $\beta$ -naphthol two molecules of base and one molecule of calcium chloride were used. In the absence of calcium chloride the yield of secondary amines was notably smaller, generally only one half. Phenyl- $\beta$ -naphthylamine crystallises in tufts of white needles, which melt at 108°. Para-tolyl- $\beta$ -naphthylamine crystallises from warm spirit of wine in white shining scales, melting at 102° to 103°. With acetic anhydride yields the crystalline compound—



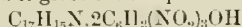
whilst by the action of benzoyl chloride the compound—



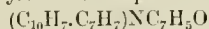
is obtained. Para-tolyl- $\beta$ -naphthylamine gives with bromine silver-white needles of the compound—



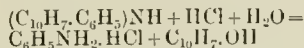
which melts at 168° to 169°. Phenyl- $\alpha$ -naphthylamine, previously prepared from  $\alpha$ -naphthylamine and aniline hydrochloride, forms white crystals, melting at 60°. Paratolyl- $\alpha$ -naphthylamine forms white crystalline tufts which melt at 88.5° to 79°. Orthotolyl- $\beta$ -naphthylamine remains persistently as an oil, but crystallises from petroleum spirit in bright scales melting at 95° to 96°. With picric acid it gives the compound—



which crystallises in reddish brown needles, and is only moderately soluble. Benzoyl chloride and orthotolyl- $\beta$ -naphthylamine yield the compound—



melting at 117° to 118°. Orthotolyl- $\alpha$ -naphthylamine forms small white shining needles melting at 94° to 95°. The data contained in this paper show, as already indicated by previous investigations, that  $\beta$ -naphthol enters into reactions with considerably greater facility than its  $\alpha$ -isomer. All the amines described gave colour reactions with nitric acid, as well as with potassium dichromate and sulphuric acid, in the case of the  $\beta$ -naphthyl compounds varying from yellow to brown, with the  $\alpha$  compounds green and greenish blue. The phenyl-, paratolyl-, and orthotolyl- $\beta$ -naphthylamines were decomposed by concentrated hydrochloric acid at a temperature of 240°,  $\beta$ -naphthol and aniline or a toluidine being formed, e.g.—

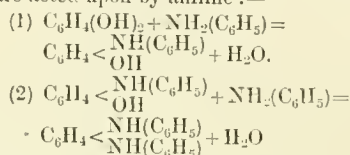


The author has been unable to confirm the statement that naphthalene and naphthylamine are formed by the action of concentrated hydrochloric acid on phenyl- $\beta$ -naphthylamine. A. S.



*Action of Aniline on Resorcinol and Quinol.* A. Calm. Ber. 16, 2786-2814.

On heating resorcinol or quinol with calcium chloride and aniline hydrochloride to 250°, or with aniline hydrochloride to 300°, metoxydiphenylamine and paroxydiphenylamine are formed respectively, the yield being very satisfactory. It is of special interest to find that resorcinol and quinol when heated with aniline *per se* react in an analogous manner. The reaction, however, goes beyond the monoxy bodies, though only in a small measure, and causes the formation of diphenylmeta- and diphenylpara-phenylenediamines. The yield of metoxydiphenylamine under normal conditions amounts to 70 to 80 per cent.; that in the case of the isomeric para- compound to 80 to 90 per cent. of the theoretical quantity. When the preparation of the diphenylated phenylenediamines is sought for it is best to use a mixture of zinc chloride and aniline hydrochloride with an excess of calcium chloride and aniline hydrochloride. The reaction takes place at 200° to 210°. The yield is high, although a small amount of the monoxy body is still present. The following equations explain the changes which occur when resorcinol and quinol are acted upon by aniline:—



The primary derivatives are distinguished from the secondary by their solubility in dilute alkalis and acids. They are precipitated from the former solutions by acetic acid, and from the latter by acetates. The two oxydiphenylamines can be distilled with ease by means of superheated steam, whilst the distillation of the phenylated diamido compounds requires a very high temperature. Zinc dust reduces the oxydiphenylamines at a moderate red heat, diphenylamine being formed besides aniline. *Metoxydiphenylamine*: White pearly laminae. Melting point, 81.5° to 82°. It is soluble in boiling water, readily soluble in alcohol and benzene. The following derivatives have been prepared:—

The hydrochloride  $\text{C}_{12}\text{H}_{11}\text{NO} \cdot \text{HCl}$   
The sulphate  $(\text{C}_{12}\text{H}_{11}\text{NO})_2 \cdot \text{H}_2\text{SO}_4$   
The barium salt  $(\text{C}_{12}\text{H}_{11}\text{NO})_2 \cdot \text{Ba} + 5\text{H}_2\text{O}$

The hydrochloride and sulphate form needles. Both are decomposed by water. The barium compound crystallises in white laminae resembling benzidine in lustre. *Diphenylmetaphenylenediamine*: Colourless flattened needles. Melting point, 95°. Is sparingly soluble in cold alcohol; readily soluble in hot alcohol, cold benzene, and ether. The following derivatives were prepared:—

The hydrochloride  $\text{C}_6\text{H}_4(\text{NHC}_6\text{H}_5)_2 \cdot \text{HCl}$   
Acetyl compound  $\text{C}_6\text{H}_4(\text{NC}_6\text{H}_5)_2 \cdot \text{C}_2\text{H}_3\text{O}_2$ . M. pt. 163°  
Benzoyl compound  $\text{C}_6\text{H}_4(\text{NC}_6\text{H}_5)_2 \cdot \text{C}_7\text{H}_5\text{O}_2$ . M. pt. 183°  
Nitroso compound  $\text{C}_6\text{H}_4(\text{NC}_6\text{H}_5)_2 \cdot \text{NO}$ . M. pt. 102°  
The hydrochloride crystallises in fine needles, and is decomposed like the corresponding compound of the monoxydiphenylamine. The acetyl and benzoyl derivatives form colourless crystals. The nitroso body crystallises in yellow needles. By treating a small amount of the nitroso compound with concentrated sulphuric acid a deep violet-blue colouration is resulted. *Paroxydiphenylamine*: Forms spangles or laminae. Melting point, 70°. Is sparingly soluble in petroleum spirit and cold water; dissolves in hot water and cold alcohol, and is readily soluble in hot alcohol. The hydrochloride  $\text{C}_{12}\text{H}_{11}\text{NO} \cdot \text{HCl}$ , crystallises in white needles, which water decomposes. *Diphenylparaphenylenediamine*: Crystallises in colourless lustrous laminae. Melting point, 152°. Sparingly soluble in cold alcohol, more soluble in hot alcohol and cold benzene, readily soluble in boiling benzene, ether, and chloroform. Derivatives prepared: The hydrochloride and the acetyl, benzoyl, and nitroso compounds. These substances agree in constitution with the analogous derivatives of diphenylmetaphenylenediamine. They

crystallise with ease, but are more difficultly soluble. The melting points of the acetyl and benzoyl derivatives are 191.7° and 218.5° respectively. The solution of diphenylparaphenylenediamine in concentrated sulphuric acid forms with nitric acid or saltpetre a deep cherry to fuchsian-red colour. On heating paroxydiphenylamine with calcium chloride, zinc chloride and paratoluidine diparatolylparaphenylenediamine is produced, besides diphenylparaphenylenediamine. This body, which forms well-defined crystals, has been prepared by the author from quinol and paratoluidine.

D. B.

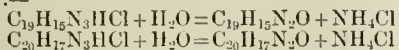
*On some Derivatives of Quinone.* R. Nietzki, Ber. Deutsch. Chem. Ges. 1883, 2092.

In order to produce nitranilic acid a mixture of 5 parts of fuming nitric acid with an equal quantity of concentrated sulphuric acid is cooled to about 8°, and to this is to be added, little by little, so that the temperature may never rise above - 3°, 1 part of finely-powdered diacetylquinol. The mixture, which towards the end of the operation becomes pasty with deposited crystals, is maintained at a low temperature until a small portion, removed for the purpose, yields a clear solution with much iced water, and gives no violet, but only a dirty brown colour with an excess of alkali. The product may be thrown upon pieces of ice, the crystals remaining for the most part undissolved. These crystals consist of nitranilic acid, which is but slightly soluble in the acid solution, but is readily decomposed by it in a short space of time. They yield a precipitate of stable potassium nitranilate with a solution of potassium chloride. It is best, therefore, to convert the acid at once into the potassium salt. To a quantity of caustic potash solution, somewhat in excess of that required to neutralise the total acid in the solution to be treated, are added a few pieces of ice, and, gradually and with constant stirring, the nitranilic acid solution. After some 12 hours the precipitated potassium salt is filtered, and may be re-purified by recrystallisation from hot water containing potassium hydroxide. In this way some 60 per cent. of the diacetylquinol is converted into potassium nitranilate. Nitranilic acid cannot thus be prepared from quinone. The potassium salt dissolves in a solution of stannous chloride, acidified strongly with hydrochloric acid, yielding after a few hours sparkling violet needles of  $\text{C}_6(\text{OH})_4\text{N} \cdot \text{H}_2\text{NO}_2$ .—W. G. M.

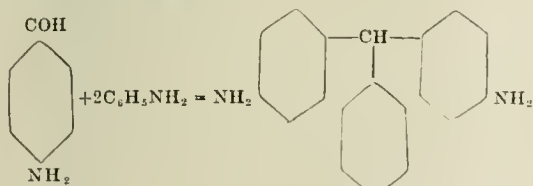
*On Chrysaniline.* O. Fischer and G. Körner. Berl. Ber. 17, 203.

THIS beautiful colouring matter has been but little investigated since the publication of Hofmann's researches in 1862 and 1869, and on account of its costliness it has not come into very general use. It occurs, together, probably, with its homologues, in company with fuchsin, and like this substance it forms a diazo compound. About two years ago, Claus observed that when chrysaniline is heated with hydrochloric acid to 160° to 180°, ammonium chloride, and a new compound, chrysophenol, derived from chrysaniline by the replacement of an amido group by hydroxyl, are formed. The striking resemblance between chrysaniline and flavaniline, as regards their fluorescence, stability, and behaviour towards fibres, made it probable that chrysaniline, like flavaniline, might be regarded as a derivative of quinoline. By the oxidation of chrysaniline Fischer obtained no oxy-acid of the quinoline or pyridine group; but chrysophenol, under the same conditions, yielded a crystalline acid which, when distilled with calcium hydrate, possessed the characteristic odour of the pyridine bases. The authors have now succeeded in proving that chrysaniline is diamidophenylacridine, both by the preparation of phenylacridine from chrysaniline and by a new synthesis of the colouring matter. Crude chrysaniline was boiled with benzene, filtered, and allowed to crystallise, and the pure substance was obtained by recrystallising the benzene compound from dilute alcohol. It was then converted into chrysophenol by heating with 3 to 4 parts of concentrated hydrochloric acid under pressure at 180°. The resulting product was dissolved in water, and poured

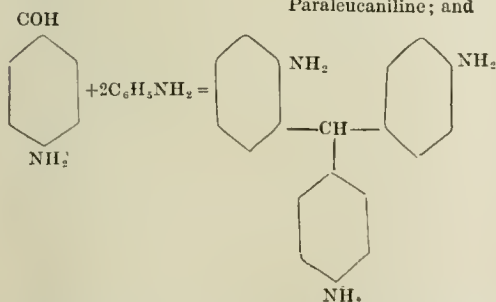
into very dilute caustic soda, when the phenol was dissolved, while the unaltered chrysianiline remained behind. The phenol was then precipitated by careful neutralisation with hydrochloric acid. It forms difficultly soluble salts with sulphuric and hydrochloric acids. The hydrochloride was recrystallised from hot water, and the phenol liberated by addition of soda. The composition of the substance was found on analysis to be either  $C_{19}H_{15}N_3O$  or  $C_{20}H_{17}N_3O$ , and its formation is thus represented:—



Phenylacridine was prepared from chrysianiline in the same way that E. and O. Fischer obtained triphenylmethane from paralencaniline, and the composition of chrysianiline was therefore proved to be  $C_{19}H_{15}N_3$ . 10 grms. of crude chrysianiline were dissolved in 50 grms. of concentrated sulphuric acid and 4 grms. of water, and nitrous acid was passed into the well-cooled solution. After removal of the excess of nitrous acid by means of a current of air, the diazo compound was poured in small quantities into 600 grms. of boiling absolute alcohol, and the greater part of the alcohol removed by distillation. The smeary impurities were precipitated with water, and the acid solution treated with excess of alkali. The mixture was then heated in an oil-bath to  $200^\circ$ , and a current of steam passed into it, the temperature gradually rising to  $250^\circ$ . A thick yellow oil came slowly over and solidified in the condenser. After recrystallisation it melted at  $181^\circ$ , and was found to be identical with the phenylacridine described by Bernthsen and Bender (*Berl. Ber.* 16, 1809). On analysis the composition of the substance was found to be  $C_{19}H_{13}N$ . The formation of chrysianiline in the preparation of fuchsine may be explained in one of three ways: (1) The paramidobenzaldehyde formed by the oxidation of paratoluidine may unite with two molecules of aniline to form the two isomeric compounds paralencaniline and ortho-diparatramidotriphenylmethane, thus:—

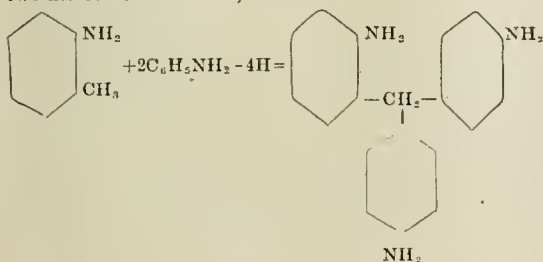


Paralencaniline; and

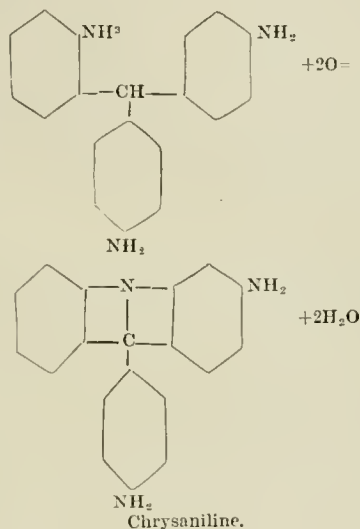


Ortho-diparatramidotriphenylmethane.

The latter compound would then yield chrysianiline on further oxidation. (2) Orthotoluidine may unite with two molecules of aniline, thus:—



(3) The chrysianiline molecule may be formed by the action of amido-diphenylamine on amido-benzoic acid, just as phenylacridine is formed from diphenylamine and benzoic acid. The second explanation is the most probable, for the authors have succeeded in preparing chrysianiline by an analogous process. Renouf prepared a substance which he considered to be orthodiparatramidotriphenylamine by the action of orthonitrobenzaldehyde on aniline (*Berl. Ber.* 16, 1304); and he obtained a brown colouring matter by its oxidation with arsenic acid at  $150^\circ$ . The authors find that at  $180^\circ$  to  $200^\circ$  chrysianiline is formed. The fused mass was boiled with water, and the bases precipitated with an alkali, and extracted with ether. On evaporating the ether, a yellow crystalline mass was obtained, which, after recrystallisation from benzene, yielded the benzene compound of chrysianiline. Assuming the correctness of Bernthsen and Bender's formula for phenylacridine, the formation of chrysianiline must be represented thus:—



S. Y.

*Improvements in the Treatment of Indigo for Use in Dyeing and Printing.* William Brookes, Chancery Lane. A communication from Thomas Holliday (Read, Holliday, and Son), Roule, Seine Inférieure, France. Eng. Pat. 2486, May 18, 1883.

THE process herein described economises both the alkali and the sulphurous acid contained in the melt resulting from the fusion of sulphonic acids with caustic alkali in the manufacture of artificial alizarin, the naphthols, and resorcinol. The solution of the melt is saturated with sulphurous acid, the hydroxy compound removed by filtration, and the filtrate, saturated with sulphurous acid, is used for reducing indigo to indigo white, by the addition of zinc dust and lime in the usual way.—R. M.

*An Improved Manufacture of Colouring Matter.* Charles Denton Abel, Chancery Lane. A communication from Messrs. Durand and Huguenin, Bâle, Switzerland. Eng. Pat. 2591, May 24, 1883. Provisional protection only.

ALPHA-NAPHTHOL is converted into its disulphonic acid by heating with five parts monohydrated sulphuric acid and two parts vitreous phosphoric acid to  $100^\circ$  C. for five hours. The product is dissolved in water and lime, the filtrate being then acidulated and treated with enough sodium nitrite to convert the sulphonic acid into the nitroso-disulphonic acid,  $C_{10}H_7(HSO_3)_2NO.HO$ . The latter is oxidised by nitric acid at the temperature of the water-bath into the nitro-disulphonic acid, which serves as a yellow dyestuff.—R. M.



*A Process of Transforming Naphthalene-disulphonic Acids into Amidonaphthalene-disulphonic Acids, and the Manufacture from these Acids of Yellow and other Dyes.* John Clayton Mewburn. A communication from Louis Freund, St. Louis, Alsace. Eng. Pat. 1069, Feb. 27, 1883.

THE naphthalene (about 44lb.) is heated with excess of strong sulphuric acid (100 kilos.) for 8 to 10 hours to a temperature of 160° to 200° C. In this reaction there are produced alpha- and beta- naphthalene-disulphonic acids which can be separated by means of the different solubility of their lime salts, but in practice the inventor appears to nitrate the mixture of acids by adding the theoretical quantity of nitric acid or a nitrate to the melt formed in the manner above described, after the completion of the sulphonation and the product has been allowed to cool. The nitration is complete in the course of some hours, when the product is diluted with water (44gals.) and the nitro-disulphonic acids reduced to the amido-disulphonic acids. The latter are converted into their lime salts and dried down. Dyes are produced by diazotising the amido-disulphonic acids, and combining their diazo derivatives with phenols, oxyphenols, their ethers or sulphonic acids, amines, etc. The inventor claims also the naphthol-disulphonic acids produced by boiling the foregoing diazo-sulphonic acids with water and the resulting diazo colours, obtained by combining them with diazobenzene and its homologues, diazonaphthalene, diazobenzene, etc.—R. M.

*New or Improved Processes for Producing Colouring Matters from Phenol.* Herbert John Haddan. A communication from the Leipziger Anilin-fabrik, Beyer and Kegel, Lindenau, Leipzig, Saxony. Eng. Pat. 3088, June 21, 1883. Provisional protection only.

THE colouring matter claimed is dinitrophenol parasulphonic acid,  $C_6H_3(NO_2)_2.HO.HSO_3(NO_2)_2$ , obtained by the action of nitric acid upon potassium mononitrophenol-parasulphonate or by excess of dilute nitric acid upon phenolparasulphonic acid. Precautions have to be taken to prevent the formation of picric acid. The salts of this dinitrosulphonic acid are used as yellow dyes, and by the reduction of the nitro groups amido acids are obtained which can be made to furnish azo compounds in the usual way.—R. M.

*Improvements in Red Colouring Matters.* Frank Wirth. A communication from Kalle and Co., Biebrich, Germany. Eng. Pat. 3095, June 21, 1883. Provisional protection only.

THE colouring matters in question are produced by oxidising one molecule of a paradiamine with two molecules of a phenolic ether. Alkyl-derivatives of the diamine may also be used, and likewise such derivatives as contain oxidising atoms in their molecule, such as nitrosodimethylaniline or dichloroquinonimide. Where no oxidising atom is in the molecule, bichromate of potash is used. As examples the authors give (1) paraphenylene (or toluylene), diamine and orthoanisidine hydrochloride; (2) paraphenylenediamine, orthoanisidine and aniline hydrochlorides; paradiamidodiphenylamine and orthoanisidine hydrochlorides; (4) Orthoanisidine hydrochloride and dichloroquinonimide; (5) nitrosodimethylaniline and orthoanisidine. The materials in examples (1), (2), and (3), are oxidised by bichromate of potash, or some other suitable oxidiser. The colouring matters (which belong to the safranin series) are red and fluorescent.—R. M.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

*A New or Improved Process of, and Machinery for, Washing and Scouring Wool.* H. J. Haddan, London. Communicated by E. Tremsal, Loth, Belgium. Eng. Pat. 2983, June 15, 1883.

WOOL treated by this invention is placed "between two endless bands or sheets situated one over the other," and

is so carried a sufficient number of times through various washing and "grease-extracting baths." The object of this manner of working is to avoid "the felting, turning, and interlacing of the filaments" of wool which are apt to take place when it is agitated in a washing or scouring bath.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Application of Hydroxylamine for Printing.* Schaeffer. Bulletin de Mulhouse, 1883.

THE author has applied the energetic reducing properties of hydroxylamine,  $NH_2.OH$ , in order to discharge manganese brown. The hydrochloride,  $NH_2.OH.HCl$  must be used. On printing this upon a manganese ground the latter is instantly reduced to manganese chloride. A very dark indigo blue dyed on manganese is lowered to a lighter and brighter blue by the elimination of the  $MnO_2$ . In like manner nankin, chamois, and similar colours can be discharged white. It is thought that a cheap hydroxylamine would be useful for technical purposes, and the Société Industrielle de Mulhouse has offered a silver medal for a cheap method for the preparation of that compound.—J. G.

*The Fixing of Aniline Colouring Matters by Means of Metallic Sulphides.* Dingl. Polyt. Jour. 250, [4], 183-184.

BLANCHE first recognises the part played by a known sulphide as a mordant for aniline colouring matters. Sulphide of zinc or sulphide of tin precipitates the colours fuchsine, aniline-violet, Bismarck-brown, etc., upon cotton fibres. Lüssy has experimented in the same way with sulphide of antimony. He fixes the latter by impregnating the fabric with Schlippe's salt and decomposing with an acid. The precipitated antimony sulphide absorbs methylene-blue, malachite-green, rosaniline, and colouring matters of the same kind, in the colouring bath. Lüssy is in error when he says that the same action takes place in tanning with antimonious sulphide as with antimonious oxide. The latter serves for the perfect precipitation of tannin, which is the main mordant, and antimonious oxide alone cannot fix the colouring matter. In order to employ the mordant action of the metallic sulphide in steam-colouring, H. Schmid recommends the "hyposulphite" method. A metallic salt, of which the corresponding sulphide can be precipitated by the action of thiosulphate of sodium, is mixed with the latter salt and colouring matter. Upon steaming, the insoluble sulphide is precipitated along with the colouring matter. In this way in one operation it is possible to fix, by means of sulphide of cadmium, copper, lead, etc., methylene-blue, malachite-green, dimethylaniline-violet, etc. The colour obtained depends on the kind of sulphide and the colouring matter employed. Sulphide of cadmium gives a green colour with methylene-blue, but with aniline-green a yellowish green. These colours withstand the action of soap pretty well. A bright yellowish steam-green is, for example, obtained with 950 grammes of unclage of tragacanth (containing 200 grammes to the litre), 200 grammes of crystallised cadmium nitrate, 300 grammes of crystallised sodium thiosulphate, 20 grammes malachite-green, 10 grammes of acetic acid of 7° B., and 150 grammes of water.—A. H. A.

*Perthiocyanogen as a Yellow Colouring Matter for Printing.* Heinrich Schmid. Dingl. Polyt. Journ. 251, 41, 1884.

THE fact that thiocyanic acid and its salts yield, under the influence of certain oxidising agents, yellow insoluble perthiocyanogen ( $C_3HN_3S_3$ ) has long been known, and Prochoroff has recently found that perthiocyanogen may be employed as a colouring matter, and has patented the industrial application of this compound under the title of "canarin." Potassium thiocyanate is oxidised by potassium chlorate in presence of hydrochloric and sulphuric

acids, and by this treatment yields about 40 per cent. of its weight of the colouring matter. Canarin appears as an orange-yellow heavy powder, insoluble in water, alcohol, and ether, but soluble in concentrated sulphuric acid and in caustic leys. Prochoroff claims its application to the printing of wool, but as regards cotton the fixation does not at first sight appear practicable, owing to the insolubility of perthiocyanogen in the ordinary reagents. The only method of applying it in cotton printing is to produce it in the fibre. This also is attended with difficulties, as it is not easy to prevent the fibre from being attacked during the very energetic action of the oxidising agents upon the thiocyanate. The author at first advised printing with a mixture of potassium thiocyanate, sodium chlorate, and metallic salts, which by double decomposition should give rise to easily decomposed salts of thiocyanic and chloric acids. Steaming would then affect the liberation of  $\text{HClO}_3$  and  $\text{HCNS}$ , and consequent formation of perthiocyanogen. It was found, however, that on steaming, an evolution of hydrochloric acid took place, sufficient to attack the fibre in the white spaces of the patterns, causing rottenness there, although the printed portions remained intact. It appeared not unlikely that by avoiding the necessity for steaming, this difficulty might be overcome, the reaction being promoted not by steaming but by the application of some vanadium solution, or by printing over such a mixture as is described above, with a paste containing vanadium. The simplest plan, however, is to print with the mixtures detailed here: 280 parts of thickening, 5 to 15 parts of vanadious chloride solution, 100 parts of commercial crystallised thiocyanate of aluminium, 100 parts of aluminium chlorate,  $40^\circ\text{B}$ . The solution of aluminium chlorate is prepared by mixing 7,900 grms. of crystallised barium chlorate dissolved in 8 litres of hot water, with a solution of 7,900 grms. of crystallised aluminium sulphate in 5.5 litres of hot water. For the preparation of the vanadium solution 20 grms. of ammonium metavanadate are dissolved in 100 grms. of hydrochloric acid and 200 grms. of water, and reduced by means of 30 c.c. of commercial bisulphite of sodium. The mixture is warmed, and when solution is complete, and a blue colour has appeared, made up to 20 litres: 1 litre of this dilute solution, therefore, corresponds to 1 grm. of ammonium metavanadate, and is thus the same as that employed in the production of aniline black. The new yellow rapidly develops in the oxidation or drying chambers such as those designed for aniline black production, or the time requisite may be reduced from 24 or 12 hours by the employment of Mather and Platt's continuous aniline black aging machine. In both cases an even brilliant yellow is obtained, and the fabric is not weakened. A remarkable property of perthiocyanogen yellow is its stability. Neither concentrated alkalis or acids destroy it or dissolve it out of the fibre, and by nitric acid its tint is simply rendered lighter. Bleaching powder and boiling soap solution have no action upon it, and air and light alter it but slowly. Another characteristic of perthiocyanogen yellow is its property of acting as a mordant to the aniline colours such as methylene blue, aniline green, red, violet, and, in short, to that class whose members are constituted as amines. This reaction cannot be ascribed to the presence of sulphur in the fibre, as it still takes place after the sulphur has been extracted. Neither is it to be ascribed to a formation of hydrocellulose, since the fixation of the aniline colours takes place when the perthiocyanogen yellow has formed, whereas it was shown above that the rotting of the fabric, due to the formation of hydrocellulose, was invariably observed in the blank spaces of the pattern. As might be anticipated from its marked deoxidising powers, wool cannot be printed with the above mixture. In this case the yellow does not develop either by steaming or drying.—W. D. B.

*Thickenings, Resists, and Discharges, for Calico-Printing, as employed principally in England.* By Robert Bourcart. Monit. Scient. xiv., 507.

#### SECTION I.—THICKENINGS.

(1) Wheat starch. (2) Mixture of wheat starch and

British gum. (3) Light and dark British gum. (4) Gum tragacanth. (5) Mixtures of starch and gum tragacanth. (6) Foreign gums, such as gum senegal. (7) Mixtures of foreign gums and pipeclay. (8) Mixtures of British gum and pipeclay. (9) Flour. (10) Mixture of flour and potato starch.

1. *Wheat Starch*.—The manufacture of this product is ably treated on in Würtz's Dictionary, in an article by Professor Schützenberger. It may be classed with flour as the cheapest thickening agent. The advantages attending its use are the following: Low price, the whiteness of the paste, the small quantity of solid matter contained in a given volume of paste. When working wheat starch the following points should be borne in mind as conditions to be avoided: (1) When made too long beforehand starch paste becomes sour, loses plasticity, evolves water and ammonia, and becomes very thin. (2) Much acetic acid must be avoided in a colour containing starch, as even weak acids prevent the paste from forming properly, especially on heating. (3) Colours containing starch should not be used for printing large surfaces, as the paste is too elastic.

2. *Mixtures of Starch and British Gum*.—These are very much used, and by mixing the two substances in various proportions, suitable thickenings are obtained for the different colours. Light British gum should be used, as this contains starch but little altered and is cheaper than the dark variety. Formerly the boiling was done over the naked flame, this not rarely giving rise to considerable irregularity from overheating, especially when the paste is thick. At the present time the heating is done by means of steam, in pans furnished with a jacket, and when the paste has become homogeneous cold water is run in instead of steam. The mixing is mostly done by means of mechanical agitators furnished with a double motion of revolution and translation, the agitator being kept acting until the paste is quite cold; without this the paste will not be homogeneous. For the testing of starch and British gum a trial colour should be made in the laboratory, half a litre being quite sufficient, printed, and then compared with a fent done with a standard sample of the same thickening. To test for the presence of silicious sand, boil with excess of dilute  $\text{HCl}$ ; this dissolves the starch immediately and gives a clear solution. Filter and then weigh the sand which is always found in starch and the different kinds of flour. If there be too much sand damage is done to the rollers and the action of the doctor is interfered with. White starch occurs in commerce as large masses, at first sight resembling crystals, and this form is to a certain extent a guarantee of its purity, for the following reasons: (1) When potato starch is mixed with wheat starch the former prevents agglutination and causes falling to powder. (2) The small quantities of gluten retained by the starch cause its particles to adhere. (3) Impurities such as lime, inorganic salts, etc., intended to whiten mouldy starch which has become gray, or added for weighting purposes, interfere with the interadherence of the starchy particles. British gum is sold as an impalpable powder of a more or less brown colour. Sometimes manufacturers mix over-burnt starch with that which has not been heated at all. This fraud is detected at once on printing, or else on inspection with the microscope. Concentrated alkalis thicken very much starch and British gum pastes by enormously swelling the granules. This takes place in the cold, and the same phenomenon is observed with mixtures of starch and water which have not yet been boiled. Use is sometimes made of this property for resists, for alkaline red mordant (aluminate of soda), and for thickening colours like natural indigo (steam).

3. *British Gum*.—This is a calcined starch occurring in light and dark varieties, and obtained by subjecting starch to various temperatures. It is distinguished from dextrine by being but little soluble, and giving a paste when heated with water. Its advantages and disadvantages, of a different nature to those of starch, are the following: Less susceptibility to the action of acids, it being quite allowable to heat this substance with a large excess of acetic acid for a long time; the paste is very smooth, and keeps a long time without breaking or



becoming thin; it forms an excellent thickening for grounds and blotches, being less elastic than starch, and thus filling up the parallel lines in the engraving. As disadvantages may be mentioned—price, brown colour, and larger quantity of British gum required than of starch for the same thickness of paste.

4. *Gum Tragacanth*.—This is the dried juice of an exotic shrub of the species *Astragalus* and possesses a very great thickening power, 20 grms. per litre being sufficient to give body to a printing colour, whereas 125 of starch or 200 grms. of British gum are required for the same effect. Gum tragacanth occurs in flattened drops, hard and of fibrous structure, sometimes bleached by chlorine to improve the colour. Alone this gum is almost solely used for thickening albumen colours as these should be washed but little, and hence require a thickener containing but little solid matter and giving a perfectly transparent paste. Much of this gum is used in mixture with starch. Since gum tragacanth is difficult to dissolve even on continued heating, it is usual to prepare a stock of standard paste containing 60-70 grms. per litre, and this is then used as required. When once properly prepared it keeps a long time without alteration. Mixtures of tragacanth and starch are exceedingly useful, being thicker than those made with starch alone, and yet more unctuous. They keep a long time and are gaining ground, especially for light designs and finely-engraved patterns.

6. *Foreign Gums*.—These are so termed to distinguish them from British gum, and consist of products obtained from the various trees of the species *Acacia*. Their properties differ but little, and they are mostly classed by quality. These gums occur in commerce as yellow or reddish-rounded fragments, opalescent and with a resinous fracture. The less dust and vegetable debris is present the better. Solution takes place easily in hot water, and extensive use is made of these substances in printing. The better qualities are used for thickening light and delicate shades, such as light alizarine, pinks, or light shades of aniline colours. Grounds on which patterns of a deeper shade are to appear are got in this way, as these gums insure great regularity of shade, owing to the presence of much acetic acid being allowable, and this prevents the weakening of the colours before printing, by retarding combination with the mordant, combination taking place only on steaming. Large quantities of these gums are required, 300 grms. per litre giving only a thin paste. A great advantage lies in the transparency and also the brilliant appearance of the printed portions. Medium and low qualities are mixed with pipeclay and used as resists, as, e.g., with dyed indigo. The salts intended to produce the white are incorporated, and the mechanical action of the gums and clay is then of considerable assistance.

7. For resists to be used with a chemie bath the above-mentioned mixed thickening is again used in conjunction with oxalic acid, and it is found that gums stand the action of strong acids much better than other thickening agents. These so-called foreign gums seem to contain metals of the alkaline earths in combination with weak acids. When thickening alizarin pinks with gum senegal, the addition of acetate of lime may be dispensed with, this being absolutely indispensable when mixing for pink with starch. Mixtures of foreign gums and starch are not workable, as pastes made thus soon become fluid again.

8. *Pipeclay*.—This is never used alone. Its use in conjunction with gum has already been mentioned, and often a mixture has been made with burnt starch, for citric resists with alumina or iron acetate mordants.

9. *Flour*.—This is used for thickening alumina or iron mordants, as the gluten contained cannot injure the colours, since all it does is to fix the metallic oxides in the cloth, and everything else is removed in the dunging, etc., operations. Potato starch, being cheaper than wheat starch, is sometimes mixed with flour. Rice starch and maize starch (corn starch) are not much used for thickening, but all the more for finishing with kaolin, a little gelatine being added for stability.

#### SECTION II.—RESISTS.

These invariably consist of pastes, made up with

various drugs and thickenings, which are printed on the cloth to prevent other colours being fixed at certain places. They may be divided into resists for white and coloured resists—e.g., those containing a different colour to the one it is desired to prevent being fixed. For each class of printing colours there is a different resist, made with the object of annihilating the power that this kind of colour has of fixing itself on the fibre. We have mechanical and chemical resists, the first class being represented by pipeclay. The second class is very large, and much in use.

*Resists with Alumina or Iron Mordants*.—These are very important resists, especially in conjunction with acid acetate of alumina, which is also termed red liquor, and is one of the most useful mordants in dyeing, as it serves to fix not only alizarin for turkey-red, ordinary alizarin red, dyed pinks, and such colours, but also when used with tannin serves for all anilines, such as methylene-blue, malachite-green, etc., and finally for wood colours. Iron pyrolignite is a similar mordant, being used either alone or else mixed with red liquor for all shades in which alizarin is the colouring matter—the scale of tints running through black, violet, chocolate, brown in various shades, and finally the red of the pure alumina lake. In order to understand how to prevent these mordants being fixed it is first necessary to discuss the manner of their fixation. The alumina and iron mordants, when printed on the cloths, are dried on tins, or else in hot rooms, and then passed into the warm moist atmosphere of the Crum oxidising chamber. The heaps of cloth are then exposed to the ordinary temperature for 24 to 48 hours. In this way the acetates are decomposed into insoluble oxides, and acetic acid, which evaporates as fast as it is liberated or is removed in passing through the subsequent becks. It is now easy to understand how the alumina or oxide of iron may be prevented from being fixed by means of a resist. It suffices to print a sufficient quantity of a non-volatile acid, such as citric, tartaric, or oxalic, to avoid the fixation of the oxides. These are, indeed, temporarily formed, but combine again to soluble salts (citrate, tartarate, etc.), which are removed by washing. Often lime juice is used as a resist, as it is cheaper than crystallised citric acid, and works quite as well. Citric acid is better than tartaric acid. The former does not form an insoluble compound with the lime frequently contained in thickeners. Tartarate of lime clogs the engraving on the roller, and prevents good printing. Resists like the above, being very acid, must be thickened with British gum, for starch cannot stand strong acid at all.

*Alumina Resist with Iron Mordant*.—This is based on the property possessed by stannous chloride of preventing the iron of ferrous pyrolignite from becoming insoluble without opposing any obstacle to the fixation of the alumina of red liquor. Print a mixture of red liquor and stannous chloride, dry, then print on the pyrolignite of iron, which will only be fixed where the resist has not been printed. In this manner we may obtain plain violet grounds, or grounds made up of lines parallel or otherwise, with floral or other patterns, not covered with violet.

*Resist in Orange or Yellow with Iron Mordant*.—Iron mordant is sometimes resisted by means of a mixture of sugar of lead and citric acid. Here the iron is not fixed owing to the citric acid, and the lead is rendered insoluble either by preparing the cloth with sulphate of soda before printing or else by means of baths of sodium sulphate and phosphate before dunging. The yellow is formed after dyeing up in alizarin by a bath of bichromate of potash, yellow lead chromate being produced, which may be turned orange by lime water.

*Resists with Dyed Alizarin and Anilines Fixed in the Resisted Parts*.—Steam aniline colours are got up as follows: Print a thickened paste of the colour, tannin and acetic acid, and dry and steam for an hour. As a rule, the printing is done on cloth prepared with stannate of soda and dilute sulphuric acid. When the colour is dried on the cloth and then steamed, the acetic acid is volatilised and an insoluble double tannate of tin and colour is formed. For the ease in hand, we cannot employ a tin prepare for the cloth, since when dyeing up the reds the

whites would come up pink, and the blues and greens would have no brilliancy. To avoid this taking place, pass the cloth through a tartar-emetie bath after steaming. The antimony combines with the tannin in the colour, and thus fixes the blue or green sufficiently well to stand dunging, red dyeing, brightening, soaping, etc., etc. Aniline colours intended to resist the alizarin or iron mordant, must contain a fixed organic acid, such as oxalic or tartaric, besides the above-named ingredients. Citric acid should not be used, as citrate of alumina decomposes on steaming, and yields insoluble alumina. The same resist may be worked with steam colours. As an example we may take steam alizarin red, *e.g.*, a printing colour containing alizarin mixed but not combined with mordants. After printing, the alumina lake is formed on steaming. Give a slight soaping to remove thickening and impurities, wash in running water and dry. The resist anilines will completely prevent the lake from forming, and yet will give insoluble tannates. Their colour comes out perfectly when all alizarin has been removed by soaping.

**Resists with Aniline Black.**—These are almost as important as those with mordants, and indeed large quantities of goods are turned out with aniline black grounds and resists in white, red, pink or other colours, being fixed by dyeing on a special alumina mordant. Aniline black being a very acid colour, it only develops on the cloth when still very acid, and hence the best means of resisting it is to print on a neutralising agent or a reducing agent, as *e.g.*, stannous chloride.

**White Resist.**—This is worked with a paste of burnt starch and caustic soda, or else the carbonate. Sometimes citric acid, or rather lime juice, is also added.

**Red and Pink Resist.**—Use is here made of the so-called alkaline red mordant, *viz.*, a solution of hydrate of alumina in caustic soda. When aluminate of soda is dried by prolonged exposure to air it is decomposed into caustic soda and alumina hydrate, which take up CO<sub>2</sub> from the atmosphere and are converted into carbonates. Print the alkaline mordant, dry, pad with aniline black not yet developed. The hydrochloric acid of the black combines with the soda of the aluminate of the mordant, the alumina is precipitated and fixed, but the black will not form, as the mixture is not acid enough. Give a few minutes in Mather and Platt's aging machine, hang for two days, wash and dung as usual, and then dye up in alizarin or else pass through tannin for anilines. If several strengths of mordant are to be printed do not let down simply with neutral starch paste, but add the white alkaline resist mentioned above, otherwise the black will not yield.

**Resists with Dyed Indigo.**—For white print a solution of copper sulphate thickened with gum and pipeclay. The copper sulphate oxidises immediately all dissolved and reduced indigo, which comes in contact with the fibre, and thus avoids entrance and fixation, the pipeclay only acting mechanically. For yellow, mix sugar of lead with the bluestone. When dyeing in the strongly alkaline vat the lead is precipitated on the fibre, a bath of dilute hydrochloric acid gives the slightly-soluble lead chloride, and a passage through bichromate of potash yields the required yellow chromate of lead, which can be changed to orange by lime water. Stannous oxide resist is much used with steam methylene-blue. The oxide of tin is prepared by precipitating stannous chloride with carbonate of soda, washing the precipitate on a woollen filter, and thickening with starch or otherwise.

#### SECTION III.—DISCHARGES.

This is another means of altering styles for printed goods, and is of great importance. We have both white and coloured resists, the former being produced by action on the colour and the latter by acting on the mordant alone. Both have as their object the destruction of the colouring matter or the mordant already fixed on the fibre, and this without injury to the strength of the fabric.

**Discharges on Turkey-Red.**—Turkey-red is an alizarin red of a shade identical with that of alizarin reds dyed by the ordinary process. It differs, however, from the latter by an extraordinary fastness, which allows it to stand the action of boiling alkaline leys. It is the

alizarin-alumina lake, more or less combined or mixed with oils altered and rendered drying, which protects the colouring matter from the action of chemicals. In this case, discharges are worked by means of a bath of bleaching liquor. As long as this is neutral, or slightly alkaline, the red keeps very well, but is immediately destroyed when the beck becomes acid; for while it is merely necessary to print tartaric acid, thickened with pipeclay and gum, before passing into concentrated bleaching liquor, made alkaline with lime-water, a thorough washing in running water is essential to free the cloth from the liquid, which otherwise would soon decolourise it in contact with the air. For yellow add a lead salt to the discharge mixture for white, and then pass through potassium bichromate solution. For blue it is usual to employ Prussian blue, this being the only blue which will stand chlorine and acids. A green discharge is got by means of a mixture of Prussian blue and a lead salt. For black it is not necessary to previously destroy the red. As a rule the blacks are obtained by superposing Prussian blue and ferrous sulphate on the red. The sulphate of iron yields the hydrate, and the combination of blue, red, and brown, produces a black. Logwood extract is also added, and this is fixed in the chlore bath, either by combination of the iron precipitated from the ferrous sulphate or else by being rendered insoluble by the lime in the liquor.

**Press Discharge on Turkey-red (Bandanna style).**—Although almost of purely historical interest, this style is perhaps worth mentioning. The dyed cloth, in layers of 12 or 14 pieces, was clamped in a hydraulic press, in which top and bottom plates were perforated to pattern. A mixture of muriatic acid and bleaching liquor, practically forming chlorine water, was forced through the perforations by means of a pump. After slackening the pressure, that part of the pieces just discharged falls into a trough of water; following portions are consecutively discharged in the same manner, following the first portion into the water-trough, and so on until the entire lengths of the pieces are finished. At present with improved machinery the work is done more quickly by printing on the acids, and then passing through the bleaching liquor.

**Discharges on Vat Blue.**—This process is based on the action of energetic oxidising agents on indigo in an acid medium. Theoretically we have here something similar to a discharge on Turkey red, but in practice the analogy does not hold. Neutral chromate of potassium is printed on the indigo dyed cloth, the fabric is dried, and then passed through a mixture of dilute sulphuric and oxalic acid, not strong enough to injure either the blue or the fibre, but strong enough to liberate chromic acid. Destruction of the blue ensues, the pattern is obtained, and thorough washing completes the operation. Coloured discharges are worked with albumen as follows: Add to the chromate of potash mixture some albumen and pigments capable of resisting strong acids, and in an impalpable powder. The acid bath in this case both liberates chromic acid and also coagulates the albumen, thus rendering it insoluble in water, acids, and soap solutions. Indifferent mineral pigments being numerous, attractive effects can easily be obtained. Thus for reds, cinnabar, red chromate of lead; for yellows and oranges, the various lead chromates; for greens, Guignet's green (hydrated oxide of chromium).

**Ferrieyanide Discharges on Vat Blue.**—This is a novel process, which allows the fixation of alumina mordant by printing on dyed indigo, destroying at the same time the colouring matter on the printed places. To obtain simply a white discharge on indigo, proceed as follows: Print a thickened mixture of ferrieyanide and caustic soda, steam, and wash. When the intention is to fix an alumina mordant, the caustic soda should be replaced by aluminatate of soda. On washing, insoluble alumina is obtained in the cloth, and it can then be dyed up with alizarin as usual.

**Discharges on Manganese Brown.**—It is usual to discharge the same series of designs on manganese brown as on vat blue, but the fastness of the colours is not as great as in the former case, for the aniline and phenol colours employed in this case fade when exposed to light; the



brown cannot stand even weak acids, and hence these prints are decolourised by perspiration. Stannous chloride, owing to its reducing action, is much used here as a discharge. Thus, by printing a thickened colour containing tin salt and eosine, the colouring matter goes on as a colourless phthaleine in combination with the tin, but, on steaming, the colour reappears, and is well enough fixed to stand a light soaping, which is, however, mostly omitted. The colours mostly employed for this style of work are catechu browns, eosines, lake of tin and Persian berries, special aniline blue resisting tin salt. The carminaphth made by MM. Durand and Huguenin, Basle, has also been tried, but is found to mark off by slow sublimation when the goods are kept in stock.—H. A. R.

*Improvements in Fixing Colours on Fabrics and in Apparatus connected therewith.* Arthur William Kirk, (J. M. Kirk and Sons, Halifax.) Eng. Pat. 3422, July 11, 1883.

IN the ordinary process of dyeing the dyed goods after being thoroughly washed are placed on a table and passed over a series of hollow rollers heated internally by steam. "With fabrics thus dyed, and more especially lustre goods employed by tailors for linings, when the tailor's iron is applied to the seams to iron the same, the colour fades or lies as the damp cloth employed by the tailors draws out the colour or colours." In order to obviate this difficulty the inventor has devised an apparatus consisting of a drying chamber fitted internally with a series of wooden rollers and ventilated by a blast of cold air. The fabric passes backwards and forwards over the rollers or remains stationary till dry, after which it is straightened over a heated cylinder and pressed or "tentured" if necessary.—R. M.

*Improvements in the Mode of Subjecting Textile Materials in Filamentous or Band Form to the Action of Dyeing, Bleaching, and other Liquors, and in Apparatus for that purpose.* Friedrich Carl Glaser, Berlin. A communication from Eugen Rummelin, Alsace. Eng. Pat. 5090, Oct. 26, 1883.

THE object of this invention is to avoid the felting or matting together of the fibres of textile fabrics which results from the operations of cleansing, bleaching, mordanting, dyeing, and rinsing as at present carried out. The apparatus described and figured in the patent consists essentially of a compound skeleton revolving reel, round which the filamentous or riband-like material can be stretched and immersed bodily in the vat containing the dyeing, bleaching, or cleansing solutions. Several of these reels can be kept going in the same bath if necessary. The advantages claimed for this method of manipulating the fabric in the bath in a stretched condition are the avoidance of felting, the treatment of considerable lengths of material without handling, the possibility of working in closed vats so as to secure uniformity of shade, and finally, the applicability of the process to existing works at a comparatively small cost.

R. M.

*Improvements in the Construction of Apparatus Employed for Bleaching, Washing, Chloring, Seouring, Soaping, Dyeing, and Dyeing Woven Fabrics.* J. Farmer, Salford. Partly communicated by A. Lalance, Mullhouse. Eng. Pat. 2871, June 8, 1883.

THIS invention "is intended to do the work much quicker, and in a more effective way, by forcing the liquor to penetrate the fibres of the cloth by a mechanical process." "The cloth is supported by hollow metallic cylinders or rollers, perforated with holes over their surface the whole width of the cloth, and corrugated lengthwise to allow the liquor to pass freely through the cloth and out or into the holes on the surface. Inside the hollow rollers or cylinders a certain vacuum or pressure is produced to force the liquor to penetrate the cloth, either by forcing the liquor inside the cylinder or roller and through its perforations through the cloth, or

by producing a vacuum inside the cylinder or roller, and thus drawing the liquor through the cloth and into the cylinder or roller." A detailed drawing of the apparatus is given.

*An Improved Method or Process of and Apparatus for Dyeing Textile Fabrics.* Henry Harris Lake. A communication from La Société Anonyme des Teintures et Apprêts de Tarase, France. Eng. Pat. 2906, June 11, 1883.

BY means of this apparatus the fabric, previously prepared by mordanting, etc., is kept continuously moving over rollers in the bath, whilst the solution of the dye is allowed to flow in from a separate cistern provided with a regulator. In this way uniformity of shade is obtained, and the amount of colour can be continuously regulated so as to meet the requirements of any particular fabric.

R. M.

*Improvements in Dyeing Silk or other Textile Fibres.* Thomas Holliday, Huddersfield. A communication from Eugene Rau, South Manchester, Connecticut, U.S.A. Eng. Pat. 2668, May 29, 1883.

THE colouring matter is chosen so as to be soluble in oil or fatty acid, or a mixture of the two solvents, the dye-bath, if requisite, being diluted with carbon disulphide, benzene hydrocarbons, or petroleum. When dyed the fabric is pressed, steamed, and the oil removed by a suitable solvent. This process is said to be especially applicable to "plushes, velvets, faille silks, ribbons, laces, and fabrics in which the lustre of the silk requires to be preserved."—R. M.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Improvements in the Salt Industry.* Dinger's Polyt. Journ. 250 [8], 363-369. *Improvements in the Boiling-down Plant of the Salt Works at Ischl.* C. v. Balzberg, Oesterr. Zeitschr. für Berg- und Hüttenwesen, 1883.

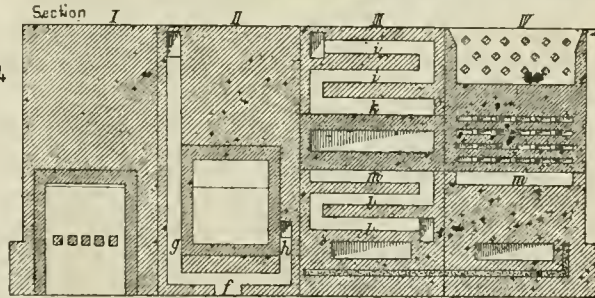
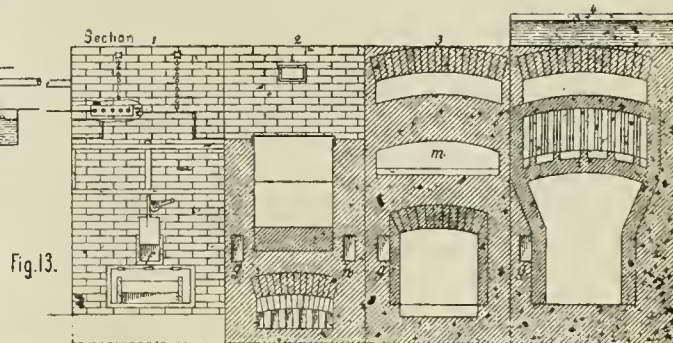
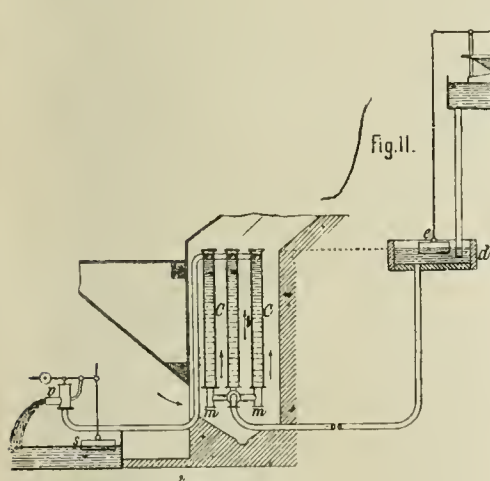
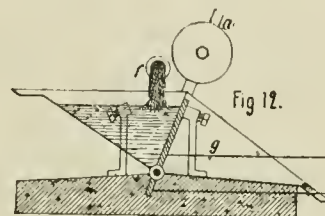
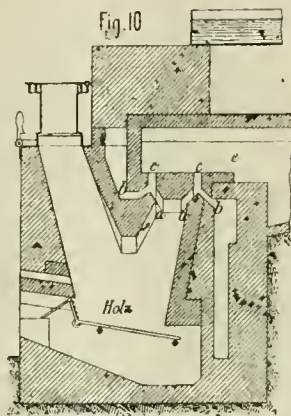
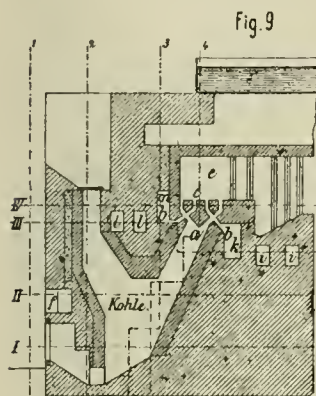
THE author calculates that if the brine indicates with the hydrometer a specific gravity showing that it is only 0.1 per cent. weaker, a loss in fuel amounting to 0.5 per cent. is incurred in evaporating to obtain the salt. The hydrometers which have been used up to the



present are not sufficiently accurate to indicate such small differences. Fig. 8 represents a new hydrometer, the scale of which shows divisions for 0.05 kilo, per hectolitre. The readings are made by means of a float, which renders it possible to estimate even 0.01 kilo. The standard temperature for which this hydrometer is graduated is 18.75° C.; and a table has been calculated by Balzberg to make corrections for any other temperature. The waste steam was used for heating up the brine in open pans; but this arrangement takes up much room,

gives a rough discoloured salt, and requires special attention, so that it has in many places been given up. The arrangement used in Ischl consists of three vessels C (Fig. 11). They are made of sheet iron, 4mm. strong, riveted together, and are connected with one another by tubes at the top and bottom. Each of these vessels is 4m. long, 1.5m. high, and has a clear depth of 0.16m.; so that the acting surface is 40 square metres. The apparatus can be easily cleaned by opening the six flanges *m* at the bottom and flushing with water. It has been found necessary, in order to work this apparatus to advantage, to give up the usual system of feeding the pans discontinuously,

which is equal to a saving of 656 kilos. of coal from Trannthal of 3,000 calories effect. In Alpine saltworks Schwind's measuring trough is used, as well as the system of measuring in large vessels of known capacity—the so-called chamber measuring. The measuring trough has been found inaccurate, and the chamber measuring takes up too much room. The apparatus represented in Fig. 12 has been found satisfactory. The centre of gravity is made movable by means of the weight *a*. In order to equalise this weight it was necessary to make the angle which the brine forms with the bottom of the vessel more pointed. In order to graduate this apparatus it is



Ischler Furnace Plant  
for the Manufacture of Salt

The depth of the brine in the pan is regulated by a copper float *s*, which by means of a valve *v* regulates the supply from the heater in such manner that the level is kept constant. A float *e* in the trough *d* regulates the supply of brine at *f*, from which place the brine has first to pass the tipping trough *g*. A registering apparatus *z* (Fig. 13, and connected with Fig. 11) thus indicates the quantity of brine consumed in the pan. If the average temperature of the steam on leaving is 43°, the brine is raised in the heater from 0° to 40°. On a daily consumption of 67,673 kilos. brine, the specific heat of which is 0.8384, this corresponds to 2,269,500 calories taken up daily by the brine,

only necessary to run a known quantity of brine through it, and to move the weight *a* up or down, according to whether the results indicated are too low or too high. The final graduating is done by fixing or taking off small metal plates on or from the weight *a*. Each of the two vessels holds 5 litres. The indicator shows every second tipping, and it is therefore only necessary to divide the number shown by the indicator by 10 in order to obtain the number of hectolitres which have passed. The scarcity of wood in the Salzkammergut induced the management to try other fuel as well. Producers were also tried, which were first built on the



system "Henpel," but were gradually altered into the forms shown in Figs. 9 and 10. As the gases were used for directly drying the salt, perfectly clean combustion was absolutely necessary. Fig. 9 shows the construction used for coal, and Fig. 10 that used for wood. The hot air meets at a right angle by passing through the slits *b* with the gases which pass through the slits *a*, and gets mixed with the latter before entering the combustion chamber *c*. The following results were obtained on analysing the gases from the original design (I.) from the furnace (Fig. 10) (II.) and from the cross-producer (Fig. 9) (III.):—

	I.	II.	III.
Carbonic acid.....	11.2 ..	14.0 ..	15 ..
Carbonic oxide .....	2 ..	0 ..	0 ..
Oxygen .....	9.0 ..	5.5 ..	4 ..
Nitrogen .....	79.6 ..	80.5 ..	81 ..

The cross-producers worked best, and yielded perfectly white salt. On burning Traunthol coal in the producer (Fig. 10), there was obtained on an average 137.3 kilos. salt for every 100 kilos. of coal; whereas the ordinary way of firing yielded only 117 kilos. salt, a saving in favour of the producer of 13 per cent. in fuel. The same producer was used for wood, but instead of the grate a plane inclined to the front was applied, and slits for the air supply. In the producers (Figs. 9, 13, and 14) the air enters at *f*; the current then divides into the channels *g* and *h*; *g* goes backward and passes through the channel *i* into the chamber *k*, where it is strongly heated and passes back into the producer. The air in the channel *h* passes through *l* and *n* into the opposite side of the burner. The feeding layer was raised to 1.15 metres. It was found that the producer gave a saving of 14.8 per cent. in wood as compared with the results obtained by the old way of firing.—J. G.

On certain Basic Sulphates. J. Habermann. Monatsch. i. Chem. 4, 787.

THE author has prepared a number of basic sulphates of nickel, cobalt, zinc, and cadmium by imperfectly precipitating the solutions of the normal sulphates with ammonia and with sodium carbonate. In their mode of formation these compounds resemble the sulphate  $6\text{CuO} \cdot 2\text{SO}_3 \cdot 5\text{H}_2\text{O}$ , described by Gmelin (vol. 3, 6 ed., p. 628) and by Pickering (*Ber.* 16, 1360). In composition, however, the analogy is not maintained. This paper is a preliminary notice of a full investigation of the subject.—C. F. C.



Improvements in or Appertaining to the Concentration or Purification of Commercial Sulphuric Acid. W. Jones Menzies, St. Helens. Eng. Pat. 3230, June 29, 1883.

THE author of this invention has "discovered that a pure hydrated sulphuric acid—that is to say, some 3 or 4 per cent. stronger than the ordinary 66 Baumé acid of commerce—can be obtained by distilling the ordinary pyrites vitriol as obtained from the Glover towers in an iron vessel or still connected with a suitable condensing apparatus, preferably a range of thin glass pipes, in which the fumes of hydrated sulphuric acid passing over from the still are condensed, and a pure hydrated sulphuric acid thus obtained. It is necessary that the acid should be of a concentration not less than that usually coming from Glover towers—that is to say, not less than 58 Baumé—and also that the arsenic shall exist in the form of arsenic acid, which is generally the case with commercial vitriol after passing through Glover towers, owing to the action of the nitric acid in the nitrous vitriol when passing through the Glover tower in the usual manner. If the vitriol to be purified and concentrated by distillation has not been passed through a Glover tower, but is simply chamber acid, it must previously be concentrated in leaden pans, with afterwards a small addition of nitric acid, or some other oxidising agent, so as to convert all the arsenic existing in the form of arsenious acid into arsenic acid. If acid of a less strength than 58 Baumé is used, there is some action on the top of the iron still, and unless the arsenic is completely oxidised into arsenic acid, it will pass over with the fumes of hydrated sulphuric acid, instead of being left

behind as an insoluble deposit at the bottom of the still. If vitriol, however, of a degree of concentration of not less than 58 Baumé be used, there is no action at all on the iron still below the surface of the boiling acid, and only a very slight action on the top of the still, when the first portions of water contained in the commercial vitriol pass off, together with some sulphuric acid, the action ceasing entirely as soon as the last traces of water have passed away." The apparatus and one mode of working are described as follows: "An iron kettle with straight sides, the metal varying in thickness from two inches at the top, where there is the greatest wear and tear, down to one inch at the bottom, has a movable iron top two inches thick, fastened on to the kettle with a tight joint. An iron elbow pipe is either fastened to the top, or forms part of it, and is arranged at such an angle that any condensation taking place in it can run back into the iron still. A small inner flange is placed at the end of the pipe to completely arrest any condensation of acid in the iron elbow-pipe being carried forward. The cooling and condensing range is constructed preferably of thin taper glass pipes, such as are now extensively used for condensing muriatic acid, fitted into each other and set at a slight inclination, so that the fumes of hydrated sulphuric acid, as condensed, shall flow down them and through the draw-off pipe to the cisterns. A leaden tower filled with flints, glazed bricks, or some other equivalent substance not acted upon by sulphuric acid, condenses the last traces of escaping sulphuric acid, and through this a small stream of water may be passed, the better to effect this object by cooling and condensing these sulphuric acid fumes. The still is provided with a feed-pipe and man-hole for cleaning out or removing from time to time the deposit of arsenic, iron, and other impurities which collect at the bottom of the iron still. A heating due from the fire-place is so arranged that neither the bottom nor top of the still are exposed to the direct heat of the fire. In working the apparatus described above, the still is first filled nearly to the top with vitriol, preferably coming hot from the Glover tower, and the fire is brought to bear upon it. At first sulphuric acid with steam passes off, forming weak acid, which can for the most part be drawn off into the cistern, or the whole of it allowed to run into the bottom of the condensing tower. As soon as the last traces of steam have passed away, pure hydrated sulphuric acid begins to distil over, and is almost immediately condensed in the row of glass pipes. This acid is drawn off into the lead cistern, which can be cooled if necessary by a coil of lead pipe with water running through it. This operation proceeds until the acid is distilled away down to the point immediately above the fire flue. The still is then refilled with acid by the feed pipe, and the operation commenced again and carried on continuously until a large accumulation of arsenic, iron, and other impurities collects at the bottom of the still, which can then be removed through the man-hole, either completely or the chief part of them, by fishing them up with an iron scoop." Another mode of working is afterwards recommended: "By thoroughly oxidising the arsenious acid and ferrous sulphate in the sulphuric acid, and heating the latter to distillation point, the arsenic and iron become insoluble in the concentrated hot acid and are precipitated in the pan or still, while acid is distilled off. The acid remaining in the still, after distillation has proceeded a short time, if withdrawn from the still without withdrawing the sediment, will be found practically free from iron and arsenic; and it will, probably, be found eventually the best plan to proceed on these lines—namely, fully oxidise the arsenic and the iron salts in the acid by means of nitric acid, boil or distill off a portion of the liquor in an iron still or boiler till the maximum strength of acid is attained, then let the precipitate settle, and decant off the hot acid."

An Improved Manufacture of Gaseous Hydrochloric Acid. C. D. Abel, London. Communicated by R. Hasenclever, Aix-la-Chapelle, Germany. Eng. Pat. 3393, July 9, 1883.

"HYDROCHLORIC acid in gaseous form, such as is specially suitable, on account of its purity, for the manu-

facture of chlorine according to Deacon's process, is obtained from aqueous hydrochloric acid by mixing therewith sulphuric acid, preferably hot, in chambers or towers of stone, earthenware, or suitable metal, the liberation of the hydrochloric acid gas being accelerated by blowing in air or stirring. The diluted sulphuric acid obtained by this process can be concentrated by evaporation and used over again."

*Improvements in the Manufacture of Chlorine.* H. A. Dufrené, Paris. Communicated by E. Geisenberger and "La Société Anonyme de Produits Chimiques, Etablissements Malétra," Paris. Eng. Pat. 3104, June 22, 1883. Not proceeded with.

HYDROCHLORIC acid is to be placed "in contact with zinc in a vessel or receiver containing plates of zinc and plates of carbon arranged alternately, so as to serve as a battery." The current generated by this battery, aided by a further current from some other sources is to be employed to decompose into its elements zinc chloride placed in "a second vessel or receiver, also containing plates of carbon and zinc arranged alternately." In the second vessel, zinc is to be deposited on the zinc plates, and chlorine liberated at the other electrodes. The zinc chloride to be electrolysed in the second vessel is to be that formed by the reaction upon each other of the hydrochloric acid and zinc of the battery.

*Improvements in the Treatment of Natural Basic Phosphates of Lime in the Leblanc Process for the Manufacture of Soda and Potash.* J. H. Johnson, London. Communicated by Crespel Brothers and Martin, Lille (Nord), France. Eng. Pat. 2990, June 15, 1883.

THE authors of this invention propose to use, in the black-ash operation of the Leblanc soda process, instead of calcium carbonate, native calcium phosphates; thus obtaining "balls" consisting essentially of calcium sulphide and sodium phosphate.

*Improvements in the Manufacture of Compounds of Alumina.* T. L. Gregson Bell, Stratford, Essex. Eng. Pat. 3388, July 9, 1883. Not proceeded with.

KAINIT or carnallite is added to a hot solution of sulphate of alumina, the product allowed to settle, and then the clear liquor decanted off. As the latter cools, potash alum crystallises out.

*Improvements in the Manufacture of Arseniate of Soda.* F. C. Blythe, Acerrington, Lancashire. Eng. Pat. 3291, July 3, 1883.

THE author of this invention proposes to obtain arseniate of soda by heating together, "in a furnace, or in a crucible, retort, or any vessel of suitable form and material," a mixture of "arsenic acid, in the solid or liquid state, with common salt, in the proportion of 1 part of salt to 2 parts of anhydrous arsenic acid, or its equivalent containing water." The chlorine of the salt is given off as HCl, which may be condensed in the usual way.

*Improvements in the Manufacture of Chromates of Soda, and in relation thereto.* E. P. Potter and W. H. Higgin, Bolton, Lancashire. Eng. Pat. 3229, June 29, 1883.

CHROME ore is to be furnace "with lime or carbonate of lime and a suitable proportion of a sodium salt, but preferably the sulphate." The product is to be lixiviated with water, and the resulting solution to be exactly neutralised by hydrochloric acid. The neutralised solution is next to be evaporated, sodium chloride and sodium sulphate being fished out as they fall. When these have been removed, the mother-liquor is to be boiled down to dryness, and the solid residuum then to be "perfectly dried, but not fused, in a top-fired mechanical pan, or by other means, care being taken to keep the temperature low," and also to keep free oxygen in contact with the salt. The product is a mixture of bichromate and chromate of soda.

*An Improved Method of or Process for Extracting Ferrocyanides from Substances containing the same.* Dr. Hugo Kunheim, Berlin, and H. Zimmermann, Wesseling, Germany. Eng. Pat. 3342, July 5, 1883.

THE inventors take "the refuse or waste products from the manufacture of gas," and desulphurise them "in the ordinary manner." The solid residue "is subsequently mixed with dry pulverised caustic lime." The mixture is then heated "to from 40° to 100° Celsius, in order to partly expel the ammonia from its insoluble combinations, the ammonia which thus escapes being condensed in the ordinary manner." By then lixiviating the mass there is obtained a solution of calcium ferrocyanide. This is then "evaporated by steam, and mixed with as much chloride of potassium as is required for the formation of ferrocyanide of calcium and potassium,  $\text{CaK}_2\text{FeCy}_6$ ." By then boiling this double ferrocyanide with potassium carbonate, "yellow prussiate of potash" is obtained. An alternative method of working is described.

*Improvements in Apparatus for Burning Small Pyrites, and Calcining Ores and other Minerals.* M. Finch, W. Willoughby, J. Willoughby, and S. Willoughby, Plymouth. Eng. Pat. 2913, June 12, 1883.

THIS apparatus is a kind of "shelf burner," the ore to be calcined in it being fed by means of a hopper to the upper shelf, and being afterwards caused to pass over all the other shelves, the solid residue of the operation being discharged at the bottom of the apparatus. The shelves, instead of being placed horizontally, like those of the Perret, Malétra, and Schaffner pyrites burners, are inclined, alternately in opposite directions.

*Improvements in Hampers or Baskets for Transporting Bottles or Carboys of Acids or Other Corrosive or Dangerous Materials, Colours, Varnishes, or the like.* H. Brunner, Widnes. Communicated by C. Garneri, Paris. Eng. Pat. 3190, June 27, 1883.

"THIS basket is built up of numerous standards of wood, thin and elastic. The base is preferably composed of two conical and concentric rings, thin, and of five to six centimetres in diameter. The standards are bound and fixed at equal distances between the two conical rings. Wooden rings placed at intervals in the height of the basket are fixed as well on the exterior as interior by means of a jointed mould, which gives to the basket the desired contour, according to the carboy or other article it is intended to contain."

*Improvements in the Manufacture of Ammoniacal Products, and in Apparatus therefor.* L. Q. Brin and A. Brin, Paris. Eng. Pat. 3089, June 21, 1883.

IN the specification of the patent 1416, of the 7th April, 1880, the authors of this invention described a method of obtaining barium dioxide by causing barium oxide to absorb oxygen from atmospheric air. Their present invention consists in passing over "barytic coke," heated to 1470° F., the mixture of oxygen and nitrogen, comparatively rich in the latter gas, which remains when some of the oxygen of atmospheric air has so been absorbed by barium oxide, thus forming barium cyanide, and in then obtaining ammonia by passing steam over that product at about 570° F.

*Process for the Preparation of Anhydrous Sulphuric Acid, and Description of Apparatus.* W. Rath, Plattenberg, Westphalia. Ger. Pat. 22118, Aug., 1882.

THE gases which leave the pyrites kiln are drawn into a condenser by means of a pump or any other arrangement, in order to free them from vapour and fine dust; they then enter a leaden tower filled with coke, through which sulphuric acid of about 66° B. is kept constantly running. The gases are here completely dried. The mixture of sulphuric acid, nitrogen, and oxygen passes then into the combining furnace, which contains vertical iron retorts lined with a fire-resisting material, and filled with a contact substance. These retorts are kept at a temperature



between bright and dark red glow, and by means of the contact substance the gases  $\text{SO}_2 + \text{O}$  combine to anhydrous sulphuric acid, which is either condensed by itself, and then mixed with hydrated sulphuric acid, or passed into the latter. If the kiln gases contain carbonic oxide and hydrocarbons (which is the case when fuel has been used for the roasting of the minerals, etc.), a special combustion furnace is placed between the kiln and the drying towers, consisting of a retort kept at a red heat.—J. G.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*On the Influence of Titanic Acid upon the Fusibility of Refractory Earths.* II. Seger, Dingl. Polyt. Journ. 83, 251, 1884.

In the *Thouindustrie Zeitung*, 1883, 243, the author communicates the results of his experiments as to the variations in the fusibility of kaolin effected by the admixture of from 5 to 10 per cent. of silicic acid on the one hand, or of an equivalent amount of titanic acid upon the other hand. The method of conducting the experiment consisted in forming the mixture into little cones, and exposing it to a temperature between those at which wrought iron and platinum melt. Tested in this manner, the kaolin employed, which contained 1.5 per cent. of foreign substances, gave a white, hard mass of well-defined form, transparent at the edges and with dead surface. A mixture of 100 parts of kaolin with 5 parts of silicic acid produced a well-defined form, snow-white in colour, somewhat porous, transparent at the edges, and giving on fracture an appearance like that of porcelain. A mixture of 100 parts of kaolin with 10 parts of silicic acid behaved as the preceding, but gave a more porous product on fracture, still more closely resembling porcelain. The cone, composed of a mixture of 100 parts of kaolin with 6.65 parts of titanic acid, had, after heating, bent over to one side, and possessed a bluish-gray glazed surface, and proved, on fracture, to have thoroughly sintered together. The mixture of 100 parts of kaolin with 13.3 parts of titanic acid fused completely to a dark bluish-gray bead of enamel. It would thus appear that the presence of titanic acid has an influence exactly the reverse of that which silicic acid exerts, the conditions of temperature being the same. It therefore seems of importance to pay great attention to the amount of titanic acid present in earths to be employed as refractory materials. The author remarks that it is not impossible that the bluish-gray colour which some clays develop when very strongly burnt is due to the presence of titanic acid, as although this colouration is usually ascribed to the presence of iron, hitherto no relation has been established between the amount of iron and the presence or degree of this bluish-gray coloration.—W. D. B.



*A New or Improved Process for Enamelling Moulded Articles of Ceramic and other Refractory Materials.* C. D. Abel, London. Communicated by A. Schierholz, Planc, Germany. Eng. Pat. 2964, June 14, 1883.

"ACCORDING to the present invention, ceramic articles, in particular statuettes, animals, flowers, or fruit, modelled in porcelain or earthenware, or in other suitable refractory materials, after being burnt or baked, are first coated or painted in the usual way with enamel colours of a syrupy consistency, and when this coating has dried somewhat, so as to be still sticky, it is thickly strewn over with small solid beads of transparent glass, which may or may not be coloured, and which adhere to the enamel coating. The moulded article is then heated in a muffle until the colours, as also the glass beads, have become sufficiently fused, whereby the ceramic article is provided with a coating consisting of the enamel colours combined with a peculiar refracting vitreous covering that imparts great brilliancy and freshness to the enamel colours, while the latter serve as a medium for uniting such vitreous coating effectually with the ceramic articles."

## X.—METALLURGY, MINING, Etc.

*Practical Results obtained from O. Springer's Double Puddling Furnace.* Dingl. Polyt. Journ. 250, 234, 1884.

EXCELLENT work has been obtained from O. Springer's double puddling furnace with regenerative firing, of which furnace a description may be found in Dingl. Polyt. Journ. 247, 329, 1883. At the works of Gebrüder Röchling, in Völklingen, 220 charges, each of 300 kilos, of raw iron were passed through both furnaces in eight shifts of twelve hours each—that is, an average of  $13\frac{1}{3}$  charges for each furnace per shift. The mean loss was 3.98 per cent., the consumption of fuel 56.90 per cent. One furnace therefore produced in the shift of twelve hours 3,960 kilos. of puddled blooms from 4,125 kilos. of pig iron, the consumption of coal amounting to 2,253 kilos. The raw iron was obtained exclusively from Minette ore—oolitic calcareous ironstone from Luxembourg—blown with reheating furnace slag. At the very best, the quality of the pig was only that of ordinary Luxembourg refined iron, and in many instances far worse. The coal employed was exceedingly poor and schistose, for it left from 50 to 70 per cent. of its own bulk of ash and cinder. The wrought iron obtained was of excellent quality and remarkably pure. The mechanical tests of the round iron of 25mm. diameter were as under: Breaking strain, (1) 37.58, (2) 38.46, (3) 37.65 kilos. per square millimetre; extension, (1) 24.2, (2) 25.6 per cent.; attenuation, (1) 36.6, (2) 43.0, (3) 43.1 per cent. It is apparent that the quality was such as to render this product specially suitable for horse-shoes, rivets, hoops, sheet iron, and tinplate. Taking into account the very poor quality of both the pig iron and of the coal employed for the production of the above, and not leaving the smallness of the amount of waste out of consideration, these results are extremely good. Should the experience of the working of Springer's furnace be the same on a large scale as above described, the puddling process cannot but undergo considerable modification.—W. D. B.

*The Manufacture of Tinplate.* Dingl. Polyt. Journ. 250, 231, 1884.

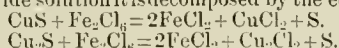
IN the *Sitzungsberichte des Vereins zur Beförderung des Gewerbfleißes*, in Berlin, F. Stoll sketches the processes adopted in the German tin plate industry. In the first place, several varieties of tinplate are distinguished according to the variety of sheet iron employed in their manufacture. These varieties are known as charcoal-plate, puddled-iron plate, coke plate, and steel plate. The first variety is still manufactured to a small extent from iron refined by wood charcoal. In the manufacture of puddled-iron plate great care must be taken in the selection of pig iron for the purpose. For coke plate, ordinary puddled iron is usually employed, since a better quality is not much in demand. Steel plate is manufactured from tough steel of different kinds. The so-called charcoal iron is obtained very hard and dense by refining pig iron and shot with wood charcoal. The tinplate made therefrom is difficult to work, but of great durability. To work the iron or steel into plates, blooms from the puddling furnace or refining hearth are flattened under steam hammers of 2,000 to 2,500 kgs. in weight, and rolled into thin rods, which are cut and made into bundles. The bundles are heated in a welding surface, thoroughly welded together, again heated in the same furnace, formed into bars by passing through grooved rolls, and cut into the size known to yield the finished plate of the required dimensions. The bars from the grooved rolls are formed into plates by passing through chilled rolls, and the plates are cut by machinery into the sizes known in commerce. By the above treatment the plates become so inflexible that previous to the polishing and cleaning process they require to be heated for from 10 to 15 hours in a well-closed muffle filled with plates. The flattening and polishing follow by passing the now softened plates between heavily-loaded chilled rolls. Before the tin can be expected properly to adhere, the plates require thorough cleansing from oxide. This is effected by the

action of dilute sulphuric or hydrochloric acid, followed by careful scouring with sharp sand. In the actual process of tinning there are five different stages. A series of five baths, with heating arrangement underneath, is built upon the same level. The baths are distinguished by the names of grease bath, tinning bath, brush bath, fine-tin bath, and grease or roll bath, the iron plate passing through the series in the same order as enumerated. In the first vessel the cleaned and scoured plate is covered with a film of melted tallow or palm oil, thence brought to the tinning vessel, freed from excess of tin at the surface by brushing with tow in the brush bath, immersed in the fourth or fine-tin bath, and, lastly, allowed to drain off any excess of metal either by standing in the final oil bath for a time, or by passing the now tinned plate through rolls fitted to this oil bath. The grease adhering to the surface at the termination of the process is removed by passing the plate through three or four boxes filled with slaked lime, sawdust, bran, or meal. The latter is the most economical in the end, as when the meal is saturated with grease it may be used as a cattle food. There now remains but the final polishing of the plate. This is effected by laying upon a table covered with flannel or sheep-skin, and rubbing the surface with a pad of the same materials. The finished plates are now sorted—reference being alone made as to their appearance—and branded as Prima-Blech (plate), W. Blech, and W. W. Blech, whilst those that appear at all defective are returned to be retinned. It has appeared desirable to do away with the oil baths, substituting baths of chloride of tin. Up to the present, however, the disadvantage of increased liability of the plates coated by the aid of chloride of tin to rust has not been overcome. W. D. B.

*On the Treatment of Rio Tinto Cupriferous Pyrites*  
Dingl. Polyt. Journ. 250, 82.

THE export ore, containing 3 to 3.5 per cent. of copper, is used, especially in England and Germany, by the vitriol maker, the roasted product being afterwards treated for silver and copper by well-known methods. Pyrites with 10 per cent. of copper is in some places roasted in heaps of about 200 tons, and charged, together with silicates and roasted copper slimes, into uniform closed hearth furnaces, 3m. high by 1m. broad and 1.2m. deep, yielding a marketable regulus containing some 40 per cent. of copper, the average daily charge worked through such a furnace being 30 tons of mixture with 16 per cent. of coke. Plumbiferous pyrites, with 5 to 7 per cent. of copper and 90grms. of silver per ton, is roasted in 200-ton heaps, and smelted with silicates in a shaft furnace, giving a regulus of 40 per cent. copper, and raw lead containing 60grms. to 90grms. of silver per ton. Pyrites with 1.5 to 2.0 per cent. of copper is roasted either in circular heaps 3.5m. high and 8m. in diameter, containing 200 tons, having a cross-shaped channel on the bottom formed by large pieces of ore and communicating with a central flue 0.3m. wide, or in 1,500-ton heaps, 17m. long by 10m. wide by 3.5m. high, with one channel running lengthwise and three transverse channels, and three flues (one at each intersection). Cavities are dug in the bottom between each two chambers (four, therefore, in the small and eight in the large heap), and in these fires of brushwood and old railway sleepers are maintained until the ore itself is burning well. The small heap will have burnt out in about two months with a consumption of 0.68c.m. of sleepers per ton of ore, the large heap in six months with 0.18 solid metre per ton. This small requirement of fuel is due to the easily inflammable nature and the rapid combustibility of the Rio Tinto pyrites and to the peculiar construction of the heaps, which favours an even and complete roasting. It is only with poor ores (1 to 2 per cent.) that the greater portion of the copper is rendered soluble by roasting. Richer ores must be subjected to a process of kernel roasting. By mixing the ore in small pieces with ferrous sulphate solution the formation of the copper sulphate in roasting is facilitated. The lixiviation of the roasted ore is conducted in tanks 30m. long by 10m. wide by 1.5m. deep. In the bottom, which slopes slightly downwards

towards one long side, are channels covered with a false bottom of planks, loosely laid, so as to permit filtration between them. In each of the lower corners of the tank is a taphole. The tapholes being closed, the tanks are filled with water to a height of a few centimetres above the ore. In about 10 hours this solution is drawn off and about ten times the quantity of fresh solution passed through, until no more copper is extracted. The solution containing 75 per cent. of the copper in the original ore passes through open channels into large reservoirs. The residue averaging 0.5 to 1.0 per cent. of copper is made whilst still moist, together with  $\frac{1}{2}$  to 1 part of rubbish, into heaps 3.5m. to 4m. high, provided with channels and flues. Heating rapidly takes place, and in six to eight weeks the freshly formed sulphate may be washed out. One and a half to two months later another washing takes place, and in 12 months the heap will be exhausted. The copper is precipitated in cemented and asphalted channels 1.5m. broad by 1m. deep, filled with pig iron. To these succeed shallower channels, also containing iron, in which the flow is more rapid. The solution must traverse 600m. of these channels in order to become decopperised. The copper is removed daily from the iron in the shallow passages by scraping with a blunt iron, from that in the deeper ones after draining. The cement copper so collected is drained in straw baskets, and carried in trucks to the roasting furnaces, where it is dried, heated, to remove part of the arsenic, cooled, and sold in sacks as oxide containing 70 to 75 per cent. Cu. The now copper free solution, after settling, is removed by pumps (composed of an alloy of Cu=80, Pb=15, Sn=5) to the extracting tanks where they are again serviceable for lixiviating. The slimes, after drying, are made into small flat cakes, air-dried, roasted in heaps, and melted with rich ore. The large consumption of iron in this process—viz., 2 parts to 1 part refined copper, or 140 parts to 100 parts cement copper—has led to precipitation by hydrogen sulphide (Dingl. Polyt. Journ., 164, 289) as in Hartmann's process. The solutions after precipitation, on account of the free acid contained, are advantageously used for a fresh extraction. Two experiments are being made at the Rio Tinto Works, with the Dötsch process, a method similar to those of Kopp and Chalandre. For this purpose, according to E. Cumenge and R. Wimmer (*Berg. u. Hutt. Zeit.*, 1883, 292), a pyrites averaging 2.68 per cent. of copper may be used without previous roasting. The copper is present as CuS and Cu<sub>2</sub>S. By treatment with ferric chloride solution it is decomposed by the equations—



This solution, for which a mixture of ferric sulphate and common salt (yielding by double decomposition ferric chloride and sodium sulphate) may be substituted, will attack only the copper pyrites, leaving the iron pyrites untouched. The ore is mixed with 5 per cent. of common salt and an equal weight of ferric sulphate, and collected in heaps from 4m. to 5m. high. These are traversed with flues and channels of dried masonry, to establish currents of air. The ferric chloride solution is evenly distributed over the surface of the heap, through which it penetrates, passing out into a reservoir, where it settles, and from which it is conducted to the precipitating vessels. By methodical washing a 2.68 per cent. pyrites will in four months have lost half its copper, or 1.34 per cent., and in two years 2.2 per cent., while in the same time, by the old roasting process and lixiviation with pure water, only 1.1 per cent. would have been extracted. With extreme care 0.32 to 0.5 per cent., or even more, copper might be washed out in ten days, and the heap be exhausted in two years. The liquors, containing little else but chlorides of copper—and that to the extent of 5 to 7 kilos. per cubic meter—are precipitated with pieces of cast iron or old rails in long reservoirs 0.75m. deep by 2m. wide, lined with gypsum and asphalt or with Portland cement. Two years' observations have indicated a consumption of iron to the extent of 1.12 ton per ton of copper (against 1.56 to 1.7 ton by the old process). The copper precipitate contains 80 to 85 per cent. Cu. The solution after this treatment contains but 20grms. Cu per cubic metre, and may, after reconversion of the ferrous chloride into the ferric salt by means of chlorine, be again used



for extracting purposes. The chlorine is obtained by heating a mixture of ferric or ferrous sulphate and common salt in air in a reverberatory furnace, air being introduced through the fire bridge. In order to decompose any hydrochloric acid which might be formed, binoxide of manganese is introduced at the back of the furnace. The chlorine passes into a flue where it meets the ferrous solution as it trickles down and converts it into the ferric salt. A four months' trial, during which time 224 tons of 85 per cent. cement copper were made, gave the following figures relating to the cost per ton:—

Lixivation—	Marks.
0.62 tons sea salt for generating Cl <sup>2</sup> and mixing in heaps at 221m.	13.65
0.2 tons sulphate of iron at 96m.	2.10
Coal, pumping, labour	51.00
	67.05
Precipitation—	
1.12 tons iron consumed	83.20
Labour	61.60
	144.80
Sundries—	
Other materials	4.20
Improvements in reservoirs	9.50
Transport of machinery, etc.	3.20
Maintenance of roads	0.96
Salaries, etc.	1.58
	22.44
<b>Total cost</b>	<b>234.29</b>

Cost of working 1 ton of commercial copper = 276m.

The amount extracted in four months was 1.34 per cent., i.e., 1 ton of commercial copper from 75 tons of ore.

	Marks.
75 pyrites at 3.20m.	240
Cost of working ditto as above	276
Transport to England, etc.	21

Total cost of 1 ton of copper = 540

But these 75 tons of pyrites would in two years yield 1.65 tons of copper; this increase of 0.65 tons must be multiplied into the working cost (276m.) and the transport (24m.); so in two years 1.65 tons of copper will have been produced at a cost of 735m., i.e., at the rate of 440m. per ton. Taking copper at 1,300m. there is evidently a gain of 860m. per ton. The composition of a sample of cement copper prepared as above was—

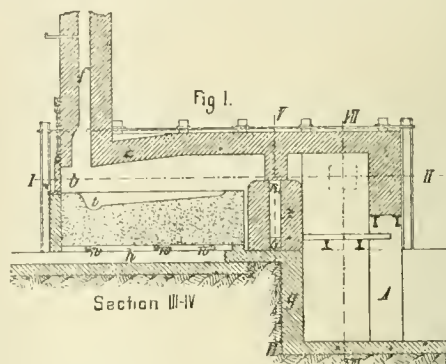
Copper	51.90
Silver	2.35
Lead	1.45
Bismuth	4.95
Iron	7.00
Antimony	0.50
Arsenic	2.95
Sulphur	5.10
Lime	0.60
Sodium chloride	0.40
Sodium sulphate	1.40
Silica	5.00
Carbon (from cast iron)	0.40
Oxygen and loss	16.00
	100.00

W. G. M.

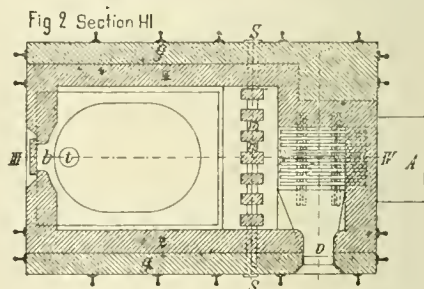
#### On the Production of Antimony and Zinc. Dingl. Polyt. Journ. 250, 123.

THE smelting of antimony is dealt with by R. Helmhaecker, in the *Berg. u. Hütt. Zeit.* 1883, 191. Antimony is nearly always obtained from antimonite ( $\text{Sb}_2\text{S}_3$ ), senarmontite ( $\text{Sb}_2\text{O}_3$ ) being found in workable quantities only in the Algerian provinces of Constantine. The liquation for crude antimony can be performed only on rich ores, those that are poorer retaining too much metal in the residue. For the crucible production of antimony, the sulphide may be fused with sodium carbonate, yielding metal and a sulpho salt, decomposable by acids to  $\text{Sb}_2\text{S}_5$ ; or the sulphide may be roasted and then mixed with raw sulphide, when a reaction occurs between them with the production of antimony and sulphur dioxide; but C. A. Hering (*Berg. u. Hütt. Zeit.* 394) remarks that this reaction does not take place unless reducing or other decomposing agents be present, the pure substances forming only glass of antimony. In the oldest process, raw sulphide is fused with scrap-iron. For smelting antimony ores in the reverberatory furnace, a solid hearth is indispensable, as the very fluid metal penetrates all crevices; and where no artificial bed is

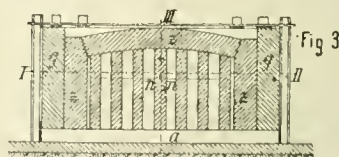
sufficiently durable recourse must be had to granite or to some similar stone. This is formed into a trough of one piece, and must be made not from the hardest stone, which will probably crack on heating, but of softer, half-weathered varieties. Unroasted ore and scrap-iron may now be placed upon the furnace bed, where the former melts readily and is decomposed. All the metal cannot be thus recovered, since a part volatilises and a part forms a double sulphide with the iron. In the process



by which antimonite is fused with sodium carbonate, the mixture froths very considerably, and is found to attack the furnace. In the third process, the partially-roasted ore, containing  $\text{Sb}_2\text{S}_3$ ,  $\text{Sb}_2\text{O}_3$ , and  $\text{Sb}_2\text{O}_4$ , is charged into the furnace with small coal, scrap-iron, and soda (or with coal and soda alone). Where it is possible 8 to 13 per cent. of coal and 9 to 11 per cent. of soda are used without the addition of iron, since with the latter the slags are less fusible and do not



entirely cover the bath of metal, and a regulus rich in iron is also obtained (*Dingl. Polyt. Jour.*, 162, 449). A furnace with a cavity on one of the longer sides, in which to collect regulus during the eliquation and the metal in the subsequent treatment, is best suited for this purpose. The great expenditure of fuel and loss by volatilisation in these processes render a method of smelting by the blast furnace desirable. Experiments in this direction have, however, at best, been only partially

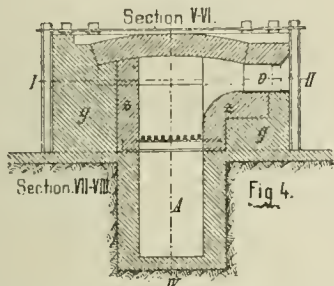


satisfactory. Helmhaecker's early experiments failed, owing to the use of a coke with some 12 per cent. of ash and of powdered oxides of antimony. He therefore made up the oxide into lumps with 10 per cent. of sodium sulphate and a little water, and charged into the furnace with 33 per cent. of charcoal. At first all went well, the charges sank regularly down, a fine rain of metallic globules fell into the crucible, and a good red heat was maintained in front of the tuyeres; but in about six hours a quantity of blackish balls of slag (consisting of  $\text{Na}_2\text{S}$  reduced from the sulphate) accumulated before the tuyeres; and from that time the temperature gradually fell at this point.

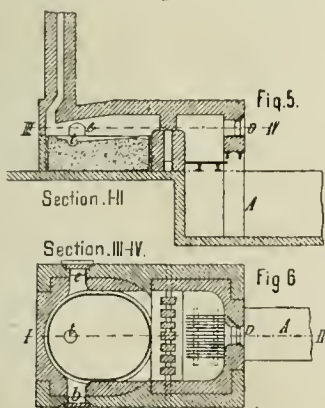
The heat, however, was maintained, and the reduction still proceeded in the upper part of the furnace. About 150 to 200 kilos. of metal were tapped during this time, the metal flowing freely and solidifying with a dark gray rough surface. No slag whatever accompanied the metal, the cause of failure being the difficulty in finding a sufficiently fusible slag; otherwise the problem to reduce antimony in the blast furnace would appear to be solved. The composition of raw antimony varies with the method of production and treatment.

	I.	II.	III.	IV.
Antimony .....	91.5	81.0	97.2	95
Iron .....	3.0	10.0	2.5	4
Sulphur .....	2.0	5.0	0.2	0.75
Arsenic .....	0.25	1.0	0.1	0.25
Gold .....	Trace	—	—	—

I. and II. are analyses of metal made in the reverberatory furnace with iron scrap, the arsenic and gold resulting

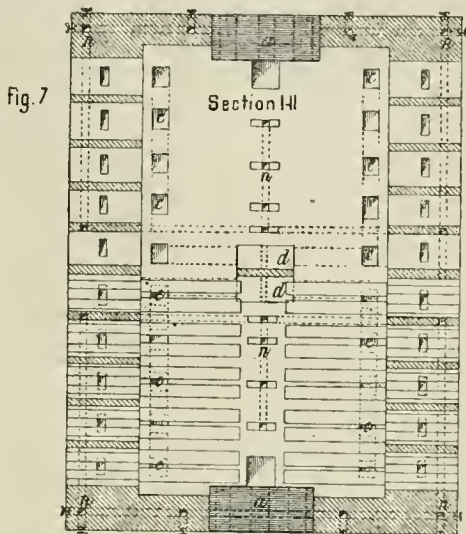


from admixed pyrites; III. and IV. were from oxidised ores smelted in the blast furnace. Figures 1 to 4 represent ( $\frac{1}{10}$ th of actual size) an antimony refining furnace, which has been practically tested. The furnace is built of firebrick walls *z*, resting on ordinary brick *g*, the whole being braced together with iron rails in the usual way. Air is admitted by the channel *a* to the fire bridge. The fire door *v* may be at the side or at the end, as in Figs. 5 and 6. The working door *b* is of firebrick bound with hoop iron, and is most conveniently situated at the end in the principal axis of the furnace, but owing to its lying thus in the direct path of the furnace gases the workmen suffer much from the smoke, and it is therefore better at the side, as in Figs. 5 and 6. Here two doors

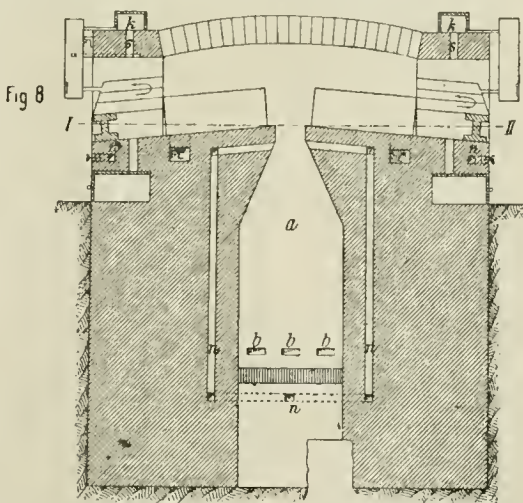


are used, the one *b* serving for the charging of the furnace and the lading of the refined metal, the other *c* for the withdrawal of slag. The bed of the furnace is constructed of one piece of hollowed granite, as described above. It stands free in the furnace, touching only the hindmost wall, and resting upon three iron wheels running on old iron rails *h*, let into the brickwork. At the deepest point in the bed is a hollow *l*, to facilitate the collection of the metal. The durability of the hearth varies greatly; some specimens crack when used for the first time, others may remain sound for a year. To avoid the expense of the granite hearths, the use of fireclay, stamped into the space between the furnace walls, has

been attempted; but although at first it is less expensive, it requires frequent renewing, since, from its greater porosity, it is more easily attacked by the soda than is the granite, and moreover it permits the metal to filter through it, and so to pass away through the walls into the ashpit. Where granite hearths are not available, the loss of metal by filtration may be obviated by stamping the fireclay into a riveted sheet-metal cylinder with ellipsoidal base (Figs. 5 and 6), which will thus retain any metal that penetrates the beds. A cheap



material which will stand to a certain extent the action of the soda is yet to be found. Steatite and magnesite have been tried and have failed, the former owing to exfoliation by the heat, the latter by insufficient solidity. When the metal to be refined is very impure, it is treated with sodium carbonate and glass of antimony, otherwise with the latter only. To prepare the glass, the  $\text{Sb}_2\text{O}_3$ , which deposits as a thick white crystalline crust on the hotter parts of the flue, is melted with liquated antimony



sulphide, and thus forms a fusible glassy dark gray or brown mass. About 600 to 700 kilos. of raw antimony are placed on the bed of the furnace heated to a moderate cherry red, fumes of the very volatile trioxide pass away, together with a part of the arsenic, and in about 30 to 60 minutes the metal will have run down. From 3 to 7 per cent. (according to the purity of the metal) of sodium carbonate is now added, sometimes mixed with powdered coke or coal. A complete covering is thus formed to the metal bath, which then fumes less, and emits here and



there small jets of flame from the surface. The temperature is raised somewhat, and the metal allowed to remain under the melted soda for the space of one to three hours (a matter to be determined by experience), until the slag becomes somewhat thick, when it must be carefully and entirely removed by drawing through the working door by a transverse flat iron with a long iron handle. To remove the iron and last traces of sulphur 3 per cent. of  $\text{Sb}_2\text{S}_3$  and 1·5 per cent. of  $\text{Sb}_2\text{O}_3$  are thrown upon the surface of the tused metal, and when these have melted, 4·5 per cent. of potassium carbonate or of potassium-sodium carbonate is added. In, at most, a quarter of an hour the refining will be complete, and the workman must proceed to the critical operation of ladling. The ladle, which is of cast iron, in the form of a hollow hemisphere, and capable of carrying 15 to 20 kilos. of metal, is riveted to a long iron handle, and supported by a chain which hangs from the roof directly in front of the working door. The cast-iron moulds are arranged upon a stone table placed before the door. The workman must dip his ladle obliquely into the metal bath, so that he may withdraw a small quantity of the glass with the metal. Part of this glass must be carefully poured into the moulds first, in order to prevent actual contact of the antimony with the iron, and then every precaution must be taken that the former is throughout protected from the air as well as from the iron by a film of antimony glass. It is only by following out these details in every particular that good star-antimony can be obtained. The stellate appearance, which is accepted as a criterion of the purity of the metal in the market, will not occur if the surface be not completely covered with glass during cooling, or if the latter solidify before the metal. Moreover, if 'eyes' of unmixed alkaline carbonate rest upon the surface of the antimony they will prevent the formation of stars at that point. The slag can generally be used a second time and contains sulphides of antimony, iron and sodium, and oxides of antimony and iron, together with silica, etc., the percentage of antimony ranging from 20 to 60. The antimony trioxide, which deposits in the flues is admixed with soot and ashes from the fuel, and is removed from the condensing chambers as seldom as possible, because the men engaged in the work are subject to headache and sickness. The men at the refining furnace work in eight-hour shifts, and must be changed at least every week, since even in that time abscesses frequently form on the legs, breast, or neck. These, however, disappear with a change of work. The furnace such as described requires 600 kilos. of moderately good coal per 24 hours, and in that time will work off two or three heats, according to the purity of the raw metal. With the proportions of materials above stated, and allowing for a loss of 20 to 30 per cent. by slagging and volatilisation, the cost of refining regulus may be taken as four to five marks per 100 kilos. K. F. Föhr, in the *Berg. u. Hütt. Zeit.* 1883-4, treats of the Boëtius zinc furnace. Each furnace has two gas generators *a* (Figs. 7 and 8), the gases traversing half the length of the furnace, then passing down the channel *d*, and thence into the flues *c* placed between the muffles. The air necessary to combustion is obtained partly through *b* directly into the gas shaft, and in part through the passages *n* in the walls of the furnace. Thus the gases are partly burnt in the upper portion of the generator, but mainly in the furnace itself by means of the air channels *n*, which are fitted at their inlet with regulating screws, so that the furnace temperature may be under perfect control. The heat is thus evenly distributed, and the muffles are therefore not destroyed so frequently. To carry off the fumes, which are so annoying to the workmen, a flue *k* passes along the top of the furnace connected with the hood of each pair of muffles. At the Lipine works, in Silesia, a movable iron hood connected with an iron flue passing just through the roof serves the same purpose, but is useless when the wind is high and causes a down draught. Glazed muffles have been used at some works, but the ordinary form is generally adopted. These cost four or five marks apiece, with an average life of 32·3 days. Dagner's condenser (*Dingl. Pol. Journ.* 236, 486), has been tried at several

works in Silesia, but the yield of metal does not appear to have been finally increased, though more oxide is obtained. The originally-increased yield gradually became less as the condensers became older and cracked, until at last it was but little greater than by the original method. One advantage of this system lies in the fact that the nozzles need not be removed in order to ladle the zinc. Cylindrical upright nozzles, as at the Hohenlohe works, collect a larger portion of the dust and oxide than those generally used, and when employed in conjunction with Dagner's condenser no zinc flame appears at the orifice, and the gases pass out through a conical nozzle on the side nearest to the furnace, and thus are far removed from the workman. A flap valve at the bottom of the nozzle allows the zinc-dust to be removed from time to time.—W. G. M.

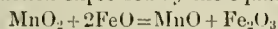
#### *On the Use of Nickel-plated Cooking Utensils.* II. Schulz, *Dingl. Polyt. Journ.* 250, 421.

THE author considers that the use of such vessels with acid fruits and the like is not quite so dangerous as Birnbaum has indicated (*Dingl. Polyt. Journ.* 249, 515, and 564). A dog, fed for twenty-nine days with rabbits which had been killed by poisonous doses of nickel, and having, in addition, 0·5grm. per diem of nickel acetate mixed with his food, was, with the exception of slight diarrhoea, commencing on the fifth day and lasting for four days, and a gradual loss of appetite, perfectly well throughout, and at the end of the time had increased in weight by 1,230grms. Broadbent has found that nickel chloride in quantities of over 0·18gr. induces vomiting. Rabbits weighing from 2 to 2·5 kilos. can take 0·5grm. of nickel acetate *once* with impunity, but 1grm. has always proved to be a fatal dose. Injected into the blood, nickel salts act as a violent poison—they possess fairly active antiseptic properties. In doubtful cases, however, the author thinks that not nickelised vessels might be used.—W. G. M.

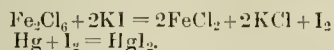
#### *On the Examination of Iron and Steel.* *Dingl. Polyt. Journ.*, 250, 413.

EXPERIMENTS by Sir F. A. Abel and W. Deering on the condition in which carbon exists in steel were reported in the *Journ. Chem. Soc.* 1883, 303. Discs of cold-rolled steel rod, originally yielding 1·108 per cent of carbon (uncombined = 0·096 per cent.) were found after hardening to yield 1·128 per cent. (uncombined = 0·035), and after annealing only 0·92 to 0·86 per cent. (uncombined = 0·052). By treatment with a solution of 99grms. of potassium bichromate and 90 of sulphuric acid per litre, a black magnetic residue—corresponding to the formula  $\text{C}_5\text{Fe}_6$ —was left. It contained nearly the whole of the carbon from the cold-rolled and the annealed samples, but only one-sixth of that from the hardened specimen. Similarly, plates from cementation steel yielded a residue of 13·25 per cent. of a carbide of iron in which the ratio between the elements was 1 atom C. : 2·65 atoms Fe. A weak solution of chromic acid gave 14·16, and a strong solution only 4·66 per cent. of the carbide. It would appear that in cold-rolled and in annealed steel carbon exists as  $\text{Fe}_3\text{C}$ , but this compound is so altered by the hardening process that it is readily attacked by chromic acid. V. Eggertz (*Berg. u. Hütt. Zeit.* 1883, 435), in writing concerning the colorimetric estimation of carbon in iron, points out that in working with artificial standard solutions the temperature of both tubes should be the same, namely, 18°, since at a higher temperature the standard appears darker than the corresponding iron solution, and this to so marked an extent that a steel which, *e.g.*, gives 1 per cent. of carbon at 18°, shows only about 0·85 at 25°. The tubes should therefore be brought to the required temperature by immersing in water at 18°. To obtain a standard steel of uniform composition, the surface of the sample must be planed or turned off. It will have lost carbon in the operations to which it has been subjected. If the standard steel approximates to 1 per cent. of carbon, clean saw files should be used for preparing the sample, as these in their hardened condition usually contain 1 per cent. and therefore there

is but slight cause for fear that the apparent composition of the specimen will be altered by the wearing of the file itself. Having once obtained a solution containing 0.1 per cent. of carbon per cubic centimetre, any desired standard may be obtained by accurate dilution. By means of this normal solution (1 c.c. = 0.01 c.) pig-iron containing over 4 per cent. of combined carbon may be treated, and will yield results within 0.1 per cent. In this case 0.05 grm. of metal should be used—it must be weighed with extreme accuracy. E. Raimond (*Revue Univ.* 1883, 460) estimates volumetrically the manganese in iron, steel, and ferro-manganese, by dissolving 3 grms. of the substance in 40 c.c. of hydrochloric acid (sp. gr. = 1.40) removing from the flame, adding 15 grms. of potassium chlorate and 20 c.c. of nitric acid (sp. gr. 1.4), digesting for a quarter of an hour until excess of chlorine is removed, and collecting the manganese-dioxide upon a filter. After washing with hot water it is retransferred to the flask in which the precipitation was effected, and into which has been introduced 50 c.c. of a solution containing 45 grms. of ferrous sulphate, to 750 of water and 250 of sulphuric acid. The reaction expressed by the equation—



then sets in, and the residual ferrous sulphate is titrated with potassium permanganate. The filtrate may be used for the molybdate of ammonia phosphorus determination. Troilus (*Berg. u. Hütt. Zeit.* 1883, 255) proposes to estimate manganese by dissolving the iron in hydrochloric acid, evaporating to dryness, digesting with nitric acid of sp. gr. 1.33 to 1.40, and precipitating with potassium chlorate. The precipitate is collected with the aid of a Bunsen pump on asbestos, washed with the strong nitric acid, and afterwards with water. It is then to be transferred to a beaker, dissolved in warm hydrochloric acid, filtered from the admixed asbestos, and heated until the last trace of free chlorine has vanished. The small quantity of iron, which was present in the precipitate is removed by ammonia and ammonium acetate. The filtrate is then made strongly alkaline with 0.88 ammonia, and precipitated with 1 grm. to 4 grms. of bromine, stirred to aggregate the precipitate, brought slowly to the boil, maintained at 100° for an hour, filtered, and weighed as  $\text{Mn}_2\text{O}_4$ . It is essential that the nitric acid used be very strong. If ordinary or diluted acid be employed with an excess of potassium chlorate, some permanganate will be formed. T. Morell's method of estimating iron is as follows: The weight of substance taken should contain about 0.2 grm. of iron when in solution as perchloride. The solution must be free from all bodies which liberate iodine from potassium iodide. It is introduced into a long-necked flask of about 300 c.c. capacity, and provided at the side with a tubulated aperture accurately stoppered, 2 grms. to 4 grms. of potassium iodide are added, and the necks closed with well-ground stoppers. The flask is then heated to 60° for a few minutes, and again completely cooled, when a weighed quantity of mercury is introduced, and the flask agitated violently in a current of carbonic acid until the solution is quite colourless. The solution is then poured off through the side tube, and the mercury washed repeatedly with water. Finally, the mercury itself is transferred to a porcelain crucible, dried with blotting paper, and weighed. The loss of weight indicates the amount of iron with the aid of the equations—



so that one part of dissolved mercury is equivalent to 0.56 parts of iron. A. Tamm (*Jern Kontoret's Ann.* 1883, 74) determines the phosphorus in iron by the following methods: 1 grm. of the metal is dissolved on the hot plate in 12 c.c. of nitric acid (sp. gr. 1.2) and evaporated to dryness. It is then heated to 200° for an hour and treated with 6 c.c. of hydrochloric acid (sp. gr. 1.19), and again heated to 200°. After re-dissolving in hydrochloric acid and removing the excess of acid, it is diluted with 5 c.c. of water, and is ready for precipitation with ammonium molybdate. Or 1 grm. is dissolved as before, but not heated above 100°, the residue being taken up in 3 c.c. of hydrochloric

acid with 2 c.c. of nitric acid. This second method gives, when metal is treated, a lower percentage of phosphorus than the former. Tamm attributes this to the solvent action upon the phospho-molybdic precipitate of organic acids formed by the action of the nitric acid upon the combined carbon, and which are decomposed at a temperature of 200°. This view would appear to be strengthened by the fact that iron ores give the same results by both methods. V. Eggertz (*Berg. u. Hütt. Zeit.* 1883, 353), in writing of his process for determining phosphorus in iron, discusses the possibility that the phosphorus, which is not precipitated by the molybdate, is present as a new variety of phosphoric acid. If it were meta- or pyro-phosphoric acid it would be converted by boiling nitric acid into the ortho condition. But this variety is apparently unchanged after boiling with nitric acid of specific gravity 1.3 and with strong hydrochloric acid. Even then precipitation will not take place. It may, however, be converted into the desired modification by causing it to unite with ferric oxide or the basic salt formed by drying the evaporated iron solution at high temperatures; and it is recommended to employ a temperature above 200°. By simple solution of the iron in nitric acid three-fourths of the phosphorus present is converted into ortho-phosphoric acid, and one quarter into the new form. Hence accurate results may be obtained with samples, not containing too large an excess of carbon and silicon, by boiling the acid solution for some time, without evaporation, and multiplying the weight of the molybdate precipitate by  $\frac{1}{4}$ . It must be remembered that samples of iron containing slag will give, on analysis, the phosphorus contained in the latter (which cannot influence the quality of the metal), in addition to that present in the iron itself. In such a case the metal may be treated with bromine, which leaves the slag unacted upon. The solution after filtration is acidified with hydrochloric acid, evaporated, dried at 200°, and the phosphorus precipitated in the usual way. The drying of the yellow precipitate is facilitated by employing a temperature of 110° to 112° instead of 95° to 100°; but the percentage of phosphorus contained must then be taken as 1.64 instead of 1.63. In dealing with ores, the results obtained after drying at 100° are equal to those resulting from the use of a higher temperature. But inasmuch as many ores contain phosphorus in the form of insoluble compounds, it may be necessary to submit them to fusion with sodium carbonate; or they may be reduced in a crucible, and the phosphorus determined in the resulting metal, since it is found that nearly the whole of the phosphorus passes into the iron under those conditions. So also in estimating sulphur, if it be present as barium sulphate, this body being insoluble, the analysis will give too low a result, but by the reduction process the sulphur will pass into the iron. When the silver plate becomes blue, owing to the presence of a large amount of sulphur, the method is not accurate. But at the same time it suffices to prove that the iron is useless for most purposes.—W. G. M.

#### Yellow and Red Oxide of Lead. A. Geuther. *Annalen* 219, 56.

By heating red oxide of lead (chrysolite, goldglätte) to its fusing point it is resolved into the yellow oxide (argyrite, silberglätte). A similar result is obtained when the salts of lead, which lose their acids at this temperature, are heated, e.g., the carbonates and nitrates. The resulting yellow oxide, after fusion and rapid cooling, solidifies into a mass of crystalline laminae. By fusing the hydrate with potassium hydroxide, and quickly cooling, the yellow oxide is obtained in a crystalline state. Likewise by introducing a boiling solution of a lead salt into boiling soda ley ( $\text{1Na}_2\text{O} + 5\text{H}_2\text{O}$ ) or hot milk of lime. Large transparent lamellar crystals, of fine lustre and yellow colour, are formed, by adding to a boiling solution, consisting of 7 parts potassium hydroxide and 14 parts of water (boiling point 110°), 1 part of finely-pulverised hydrated oxide of lead. The mixture is kept at this temperature until nearly all the lead has been dissolved, after which the solution is allowed to cool gradually. The formation of a red-coloured oxide of



lead from the hydrate begins at  $110^{\circ}$ , and in order to complete the same it is necessary to raise the temperature to  $150^{\circ}$ , and conduct the heating in a closed crucible. For the conversion of the carbonate into the red oxide a high temperature is required. To obtain the red oxide in the form of lamellar garnet-red crystals it is recommended to dissolve 1 part of the hydrate in 5 parts fused potassium hydroxide and slowly cool the mixture. By boiling the hydrate in a solution of 3 parts sodium hydroxide and 4 parts water (boiling point  $130^{\circ}$ ), red crystalline hydrated oxide of lead is obtained. The yellow oxide has a specific gravity of 9.28 to 9.36, the red 8.74 to 9.126. The former crystallises in rhombs, the latter in a tetragonal form. It is further shown that at the ordinary temperature the yellow oxide may be resolved into the red by pressure and friction, whilst to convert the red oxide into the yellow it is necessary to heat the same almost to the fusing point. By fusing the oxide with potassium hydroxide in atmospheric oxygen a compound of peroxide of lead and potash is deposited, on cooling, in the form of light-brown six-sided tables.—D. B.

*The Dephosphorisation of Iron.* Dingl. Polyt. Journ. 330, 250, 1884.

As communicated by him to *Stahl und Eisen*, 1883, 498, G. Hilgenstock has observed that a crystalline formation takes place at times in the slags obtained in the dephosphorisation of iron by the basic process. He has isolated such crystals as carefully as possible, and has found them to possess the percentage composition of 61.10 CaO, 38.14  $P_2O_5$ , corresponding to the formula for tetra-basic calcium phosphate,  $Ca_4P_2O_{14}$ . This occurrence of the tetra-basic phosphate explains, to some degree, the necessity for the comparatively large amount of lime in the basic process. Carnot and Richard, in the *Revue Industrielle*, 1883, 365, record the occurrence of transparent blue crystals in the cavities of a basic slag from Jonel. The crystals were found to correspond in their composition to that of a silico-phosphate of calcium, together with small quantities of ferrous oxide and alumina. The analytical results are as under—

$P_2O_5$ .....	29.65
$SiO_2$ .....	12.42
$Al_2O_3$ .....	2.76
CaO.....	53.20
MgO.....	Trace
FeO.....	1.80
$Mn_2O_3$ .....	Trace
	99.83

W. D. B.

*An Improved Process and Method for Making Refined Cast Steel from Inferior Wrought Iron, as well as the Best Quality of Wrought Iron.* T. Sheehan, London. Eng. Pat. 2862, June 8, 1883.

TREATMENT of "inferior wrought iron" by, per 20 tons thereof, 110lb. of "muriate of soda," 80lb. carbonate of soda, 70lb. bisulphate of soda, 40lb. sulphite of zinc, and 6 bushels of hardwood charcoal, together with alumina and "granular limestone."

*An Improved Process of Treating Lead to Impart to it the Property of Adhering to and Amalgamating with other Metals.* P. M. Justice, London. Communicated by F. J. Clamer, Philadelphia, U.S.A. Eng. Pat. 3933, Aug. 14, 1883.

"THE object of this invention is to treat melted lead" in such wise as to "so change the nature of the metal that, by a process similar to galvanising with zinc, the surfaces of iron, copper, brass, etc., may be covered with a smooth and permanent coating of lead, free from holes, and not liable to scale off." The "small holes and blisters so frequently found in" the coating of "a sheet of metal coated with lead in the ordinary way," are attributed to the molten lead absorbing gases, which are given out on cooling. "Melted lead is also thick and sluggish, consequently a coating formed in the usual way is full of ridges, the metal being very unevenly

distributed over the surface of the sheet." By adding to 100 pounds of molten lead "first three ounces of sal ammoniac, then half an ounce of arsenic, after this one ounce of phosphorus, and lastly half an ounce of borax," the lead is said to have all gases driven out of it and to be rendered limpid, with the result that sheets or other articles of iron, copper, or brass, dipped into a bath of it, become coated with it perfectly evenly.

*An Improvement or Improvements in the Manufacture of Alloys of Tungsten.* F. W. Martino, Sheffield. Eng. Pat. 3421. July 11, 1883.

OWING to the very high fusing point of tungsten, it is not practicable to form alloys of that metal with metals which fuse at very much lower temperatures, by melting it and them together. The author of this invention obtains alloys of tungsten with copper, tin, and zinc as follows: "I take a mixture of metallic tungsten and phosphide of calcium and fuse the mixture in a crucible, preferably a plumbago crucible. I keep the crucible well covered with a lid, and the contents of the crucible covered with a layer of powdered charcoal, during the fusion of the mixture of tungsten and phosphide of calcium. By the action of the phosphide of calcium on the tungsten, a regulus of phosphide of tungsten is produced. I add this phosphide of tungsten to metallic copper or to phosphor bronze and melt the mixture in a plumbago crucible, the mixture being covered with a layer of powdered charcoal. In this way I obtain an alloy of tungsten and copper, the said alloy also containing phosphorus. By the addition to this alloy of tungsten, copper, and phosphorus of an alloy of copper and tin or copper and zinc, I produce a series of alloys having such colours and possessing such ductility and malleability as fit them for the manufacture of a variety of articles, such, for example, as spoons, forks, fish and dessert knives, as well as vessels of various kinds."

*Improvements in the Treatment of Compounds of Aluminium for the Purpose of Obtaining therefrom Aluminium or Aluminium Bronze, and in the Means Employed in the said Treatment.* J. H. Johnson, London. Eng. Pat. 2888, June 9, 1883. Communicated by J. Varin, Paris. Not proceeded with.

THE author of this invention proposes to electrolyse fused double chloride of aluminium and sodium, using as negative electrodes either retort charcoal or aluminium plates, and as positive electrodes plates of "an intimate mixture of alumina and carbon." The chlorine evolved at the positive electrode is to combine with the aluminium of the alumina of these electrodes, forming aluminium chloride, by which to continually replenish the bath, and the oxygen of their alumina is to combine with their carbon, and so to be evolved as carbonic oxide.

*Improvements in the Method or Process of Treating Ores and Substances containing Antimony and other Metals for the Recovery thereof.* J. C. Butterfield, Westminster. Eng. Pat. 2945, June 13, 1883. Not proceeded with.

ANTIMONY ore is to be reduced to an impalpable powder, and then boiled, with frequent stirring, with a solution of ferric chloride of  $70^{\circ}$  T. "The perchloride of iron is reduced by the antimonous sulphide, whilst the antimony is dissolved as chloride, with separation of sulphur." From the solution of antimony chloride thus obtained, free antimony is then precipitated by scrap iron.

*Improvements in the Treatment of Tin and Terne Plates.* D. Grey, Maesteg, Glamorgan. Eng. Pat. 2874, June 8, 1883.

THIS invention consists in substituting for the palm oil at present used by manufacturers of tin plates various mixtures of linseed, resin, Rangoon, castor, and cotton oils, and especially a mixture of linseed oil and resin oil, in the proportion of 65 parts of the former to 35 parts of the latter.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*Cotton-Seed and Palm-Nut Oils.* Seifensied. Zeit. 33, 390.

ON account of the high price of tallow, these oils are almost indispensable to the soap manufacturer. Both oils are commonly used together for making hard soaps, cotton-seed oil being particularly useful in summer, on account of the large proportion of stearin (about 30 per cent.) it contains, which renders the soap more compact and capable of withstanding the action of higher temperatures. Crude cotton-seed oil has a reddish-brown colour, and when intended for exportation is usually treated with soda. The oil thus refined has a density of 0.926, and solidifies at about  $2\frac{1}{2}^{\circ}$  C. During late years cotton-seed oil has been extensively used for the adulteration of olive oil. Palm-nut oil is obtained from the palm-nut, either by pressure or extraction with carbon disulphide or light petroleum, the latter being preferable, as carbon disulphide, when not perfectly eliminated from the oil, causes serious trouble, giving both a bad smell and colour to the soap made from it. Palm-nut oil is seldom used by itself for soapmaking, as it produces a very brittle and easily-cracked soap, which combines with but little water, and thus causes the produce to be very low. Palm-nut oil is, however, of importance when mixed with other fats. In order to free ordinary refined cotton-seed oil from colouring matter it is treated with the entire quantity of ley, at  $24^{\circ}$  Baumé, required for the complete saponification of the fats to be used, and the mixture boiled. The other fats are now added, and, after addition of water, the saponification, which has not yet entirely taken place, is completed.—A. H. A.

*Neutral Soaps, for Soaping Coloured Goods.* Chem.-techn. Centr. Anz., Seifensied. Zeit. 34, 405.

IT is known that a soap is required for brightening the colour of garancin, or artificial alizarin, on printed goods, without causing change or injury. Marseilles soap is given the preference without any clear idea of what constitutes the difference between the various soaps. O. Scheurer, in the *Bulletin de Mulhouse*, says that when the soap acts on the colour, as is especially the case with olein soap, it is because the soap has been insufficiently saponified, part of the oil used being undecomposed, and free alkali (which does the mischief) left in solution, causing the reds to turn brown, injuring the violets, and spoiling the whites. He points out that although the proper quantity of alkali may be present, if the boiling of the soap is not continued sufficiently long the result will be an alkaline soap. Increased temperature and pressure very much hasten the formation of a neutral soap. Boiling for two hours, under a pressure of  $1\frac{1}{2}$  atmosphere, gives a better soap than twelve hours under the ordinary atmospheric pressure.—A. H. A.

*Fat and Grease of the Chinese Tallow-Tree.* Techniker, Seifensied. Zeit. 35, 415.

THE high price of tallow of animal origin has caused the attention of soap and candle manufacturers to be turned to vegetable fats. The tallow-tree is a native of China. It produces a fruit of which the kernel is covered with a thick layer of tallow, and contains yellowish aromatic oil, useful for heating purposes. After the fruit has been gathered, which is done in South China in July, and in the North in October, hot water is poured over it. When the water has cooled, the tallow can be taken off. It is then run into bamboo-cane moulds ready for the market. It has a green colour, crystalline structure, possesses a characteristic smell, and melts at  $40^{\circ}$ .—A. H. A.

*A Method of Freeing Tallow from the Smell of Petroleum.* Seifensied. Zeit. 39-462.

IN Germany, tallow, treated with 2 per cent. of petroleum oil, is free from duty. The writer gives the following method of freeing it from the objectionable smell of the

petroleum. The sample is placed in a cauldron, and for every 100 pounds weight about half a pound of zinc chloride, dissolved in water, is added. Steam is now passed through the liquid for from two to three hours. Instead of steam, the sample may be well boiled with a fourth of water for the same length of time.—A. H. A.

*Russian Mineral Machine Oils.* "Bakuin." By a Foreman of A. Oehlrich and Company, Riga. Seifensied. Zeit. 31, 366: 32, 378.

VARIOUS brands of oil are made corresponding to the different viscosities required in lubricants. Two qualities, known as Bakuin 1A 1A and Bakuin AA have respective densities of 0.910 and 0.905. These oils are recommended for lubricating heavy machinery, on account of their high viscosity and great power of resisting cold. The Bakuin oils generally are said to be greatly more economical than vegetable oils, and to freeze less readily. Astralin is a burning oil, which can be used in ordinary petroleum lamps, and it is said to be less inflammable and to be consumed more slowly than petroleum.—A. H. A.

*Improvements in the Method of Treating Linseed and other Drying Oils, so as the better to fit them for Use in the Arts and Manufactures.* A. Ford, London. Eng. Pat. 3193, June 27, 1883. Not proceeded with.

THE linseed or other oil treated by this invention is heated to from  $130^{\circ}$  to  $150^{\circ}$  F., solution of diacetate of lead is added to it, and into the mixture air is injected, during 10 hours. The mixture is then allowed to settle, the "foots or settlings" are separated, and on the clear part the above-described treatment is repeated, "only at a much lower temperature" than the first time. The product is said to possess "such drying powers" that paints made with it need not be mixed with "turpentine or other dryers."

## XII.—PAINTS, VARNISHES, AND RESINS.

*An Improvement in the Manufacture of Anti-fouling Paints or Compositions.* A. M. Clark, London. Communicated by C. Dubois, Marseilles. Eng. Pat. 3320, July 4, 1883.

"THIS invention consists in the combined employment of the sulpho-cyanides of copper and the arseniates of mercury in any kind of paint for marine purposes."

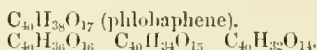
## XIII.—TANNING, LEATHER, GLUE, AND SIZE.

*Tannins of Oak Bark.* C. Ettl. Monatsh. f. Chem. 40, 512.

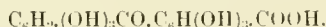
THIS is a critical comparison of the author's results with those of Böttiger and Löwe. The following is an outline of his main conclusions. The tannic acid of the oak bark occurs both as the body ( $C_{17}H_{16}O_9$ ), and as its first or semi-hydrate ( $C_{17}H_{14}O_{17}$ ), which latter constitutes the phlobaphene of the bark. On boiling this semi-hydrate with dilute sulphuric acid the second anhydride, ( $C_{17}H_{12}O_{16}$ ), is formed. The original tannic acid, on the other hand, yields, on boiling with the acid, the third anhydride ( $C_{17}H_{10}O_{15}$ ). The fourth anhydride ( $C_{17}H_{8}O_{14}$ ), was obtained by Löwe by heating the original tannic acid with either water or dilute sulphuric acid at  $110^{\circ}$  for eight days. Various compounds described by Usher, Grabowski, and the above authors are severally identified with these bodies, and thus a number of discrepancies are cleared up. In the isolation of the original tannic acid special regard must be had to the first of these anhydrides, which, in addition to being likewise present in the bark, is formed from the tannic acid in the course of its separation by means of solution in acetic ether. This acid, in fact, decomposes the acetic ether, and the resulting acetic acid exerts a dehydrating action upon the tannic acid. The author, therefore, in the last stage of purification uses ether-alcohol as the solvent. In addition to this phlobaphene, the crude tannic acid contains a terpene resin, which is easily eliminated by taking advantage of



its solubility in benzene. The sugar obtained by certain observers on boiling the tannic acid with dilute mineral acids is shown to result from the levulin present in the bark. From the number and mode of formation of the above anhydrides, together with the evolution of methyl chloride on heating the tannic acid with hydrochloric acid, the author concludes that it is a methyl derivative of a gallylgallic acid,  $C_{40}H_{36}(ON)_{13}.CO.C_6H_5N(ON)_3COON$ . The author has also investigated a tannic acid ( $C_{20}H_{20}O_9$ ) obtained from the bark of a different species of oak. This tannin gives a green colour with ferric chloride, but in other respects was closely similar to the tannic acid described above. Thus it yielded four successive anhydrides—



under conditions of dehydration exactly resembling those employed with the previous acid. On subjecting the acid to dry distillation there were obtained from 1,500 grms. 4grms. pyrocatechol, 800grms. of the pseudo-carbon, and 2·6grms. of an oil, apparently a terpene, and derived from the resinous impurity of the acid. On heating the acid with hydrochloric acid very little methyl chloride was formed, whence the presence of oxalcoholic groups was to be inferred. The reactions of the acid otherwise point to a similar deviation from a gallylgallic acid—



Lastly, the author suggests that the phenols and phenol-ethers present in wood tar are probably formed at the

finally be evaporated *in vacuo* to a density of 20° B. If it be desired to remove mucilaginous matter as well as colouring matter, four times the above-mentioned proportion of alumina must be used.

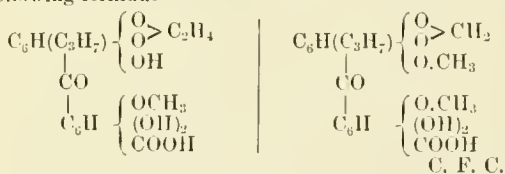
#### XIV.—AGRICULTURE, MANURES, Etc.

*On the Superphosphate from Animal Charcoal.* Prof. Fr. Farsky. Biedermann's Central-blatt. für Agrikulturchemie, 12, 449. Bericht der landw-chemischen Versuchsstation in Tabor für das Jahr, 1881 and 1882.

PREVIOUS investigations by the author had proved that the Bohemian superphosphate from animal charcoal prepared in different works contained free phosphoric and sulphuric acids and dicalcium phosphate, besides the chief constituents, monocalcium phosphate and calcium sulphate. Free sulphuric acid was only found in superphosphates which contained no tricalcium phosphate, or in poor products prepared by the admixture of richer ones with earth. Its amount ranged from 0·1305 to 1·0548 per cent. Monocalcium phosphate was found in superphosphates which had contained more than 1 per cent. of tricalcium phosphate, and in such as had been mixed with clay, lime, or calcareous earth. Calculated as phosphoric acid, it amounted to 0·0241—1·9417 per cent. Free phosphoric acid was only detected in rich products to which too much acid had been added. Coarse-grained superphosphate contained more free acids than fine ones. The amount varied from 0·2452 to 2·5000 per cent. It

State of Aggregation.	Water Added.	Loss of Soluble Phosphoric Acid in							
		6 hours.	12 hours.	1 day.	2 days.	4 days.	5 days.	7½ days.	20 days.
Coarse	Little	16·57	23·88	25·37	28·91	31·57	32·63	31·23	39·62
Coarse	More	20·17	31·83	37·23	42·10	47·28	49·34	51·99	60·28
Coarse	Allowed to dry	—	—	—	—	—	—	18·88	—
Fine	Little	20·32	31·83	35·73	40·01	45·25	—	50·89	—
Fine	More	25·12	37·00	44·12	50·71	57·92	—	68·12	—
Fine	Allowed to dry	—	—	—	—	—	—	23·33	—

expense of a tannic acid, having a constitution represented, at least in its main outlines, by one of the following formulæ—



*Improvements in the Preparation of Tannic Extracts.* J. H. Johnson, London. Communicated by E. L. P. Coëz and G. C. Coëz, St. Denis, France. Eng. Pat. 3150. June 26, 1883.

THE "tannic extracts heretofore supplied to tanners are to a certain extent rendered colourless by various processes," but these are processes by which "the colouring matter" of the extracts is "merely neutralised by chemical agents," leaving it "liable to reappear at any time under certain conditions, such as age or exposure to the air." By the present invention, the colouring matter is to be removed, instead of simply neutralised. To this end, to tannic extracts "obtained by the usual processes" oxalic acid is added, in the proportion of 1grm. to 100 litres of decoction, "for the purpose of saturating the lime of the water used in the process of extraction." Then gelatinous alumina is added, in the proportion of 250grms. per 100 litres of decoction. The alumina combines with the colouring matter present, and filtration now yields a "clear tannic extract," which must

appear that excess of acid decomposes the monocalcium phosphate with formation of dicalcium phosphate and free phosphoric acid. The author concludes that better results are obtained by the acidification of fine than of coarse raw material, and that free acid is not so likely to occur when the acid is poured on the animal charcoal as when that substance is added to the acid. *Action of Water on Superphosphates:* A sample of superphosphate was separated by a sieve into two portions. The coarser contained 11·6, the finer 10·26 per cent. of soluble phosphoric acid, but no dicalcium phosphate. The substance was placed in vessels with finely-porous bottoms, which stood in other vessels. In most cases 2—4, and later 1·2—2·4c.c. of cold water were sprinkled on the different portions. Two samples were allowed to dry, and several were not moistened at all. The latter remained entirely unaltered, while the others were acted on as above. Thus by moistening the superphosphate and allowing it to stand in the air, soluble phosphoric acid is lost. The nature of the reaction is indicated by the results tabulated below:—

Water added.	Amount of Products formed in Percentages of the Original Phosphoric Acid				
	as $CaH_4(PO_3)_2$	as $CaHPO_3$	as free acid	washed out	decomposed
Little	52·30	23·86	23·84	—	47·10
More	50·13	21·94	18·11	6·82	49·87
More	—	30·33	—	12·21	—
More	—	37·75	—	11·22	—

The mono-calcium phosphate is thus decomposed with formation of free acid and the dicalcium salt. The above results show clearly why, in the analysis of superphosphates, the samples must be treated at once with plenty of water, otherwise the percentage found would be too low. *Behaviour of Superphosphates in Moist Earth:* Every 100grms. of earth containing 7.78 per cent. of calcium carbonate and 0.12 of tricalcium phosphate were mixed with 10 grms. of 12.3 per cent. fine, or with 8.8489grms. of 13.9 per cent. coarse superphosphate, and sprinkled repeatedly with 5 or 10c.c. of water. The residual soluble phosphoric acid was estimated from time to time with the following results:—

becomes insoluble. (3) The retrogression of finer grained superphosphates is at first more rapid than that of coarser, but here, again, the difference becomes gradually smaller. (4) Since the germination of moist seeds in the earth occupies from seven to fourteen days, it follows that in calcareous earths the plants obtain their phosphorus chiefly, if not solely, in the form of the calcium phosphate soluble in citric acid. *Experiments under Glass:* Several vessels 65c.m. high, and of 1,000 square cubic centimetres surface, were filled with earth of known composition, which had been mixed with 20 per cent. of calcium carbonate. The plant employed was the Tartary buckwheat. It was sowed thickly at first, and

State of Aggregation of Superphosphate.	Water added.	Loss of Soluble Acid in Percentages of the Original Quantity.										
		3 Hrs.	6 Hrs.	12 Hrs.	1 Day.	2 Days.	4 Days.	6 Days.	10 D'ys.	14 D'ys.	18 D'ys.	23 D'ys.
Coarse	—	—	—	—	—	—	—	—	0.74	—	—	1.10
	Little	62.50	72.32	80.10	85.12	89.13	92.28	94.71	96.00	97.49	98.31	98.48
	More	60.30	67.11	71.25	80.12	81.20	87.95	89.97	91.75	95.22	97.44	98.15
Fine	—	—	—	—	—	—	—	—	1.12	—	—	1.78
	Little	70.17	79.81	84.28	88.05	90.90	92.30	94.28	96.15	97.98	98.65	98.82
	More	68.55	75.92	81.81	85.52	88.24	90.72	92.87	95.12	97.14	98.15	98.81

Thus the retrogression of the superphosphate in the earth was very rapid. It was found by analysis that acid aluminium and iron phosphates were formed by the action of the free acid on the alumina and iron compounds. They are, however, unstable, and had almost disappeared by the sixth day. The presence of free acid was ascertained by further experiments. The acid is

then weeded. The results are given in the table below. From these results the author concludes: (1) That the plants are developed more regularly, and that the crop is greater when the superphosphate is strewn by hand, than when it is previously mixed with earth. (2) That the best result is obtained when the manure is placed below the seeds. (3) That the action of the super-

State of Aggregation of Superphosphate.	Method of Manuring.	Time of Manuring.	Description of Earth.	Weight of Crops in grms.			Weight of 100 Seeds.
				Seeds.	Straw.	Total.	
Not Manured	—	—	Clayey.	26.412	13.517	39.929	1.480
—	—	—	Ditto	26.199	13.285	39.484	1.482
—	—	—	Calcareous	25.235	12.712	37.977	1.483
—	—	—	Ditto	25.507	12.569	38.076	1.480
Coarse	Mixed with earth and raked in superficially Very carefully distributed	At Sowing	Ditto	29.891	15.507	45.401	1.522
Coarse		Ditto	Ditto	32.141	16.585	49.029	1.529
Fine		Ditto	Ditto	29.215	15.511	44.726	1.525
Coarse	5 c.c. below the seeds carefully distributed	Ditto	Ditto	32.699	16.792	49.791	1.532
Coarse		Ditto	Clayey	31.723	16.352	51.075	1.525
Fine		Ditto	Ditto	33.675	16.155	50.130	1.528
Coarse	Ditto	After Germination	Calcareous	32.615	16.207	48.822	1.528
Fine	Ditto	Ditto	Ditto	30.170	15.811	45.981	1.529
Coarse	Ditto	Ditto	Clayey	34.750	16.564	51.314	1.527
Fine	Ditto	Ditto	Ditto	33.778	16.500	50.278	1.528

rapidly neutralised by the excess of salts in the earth, forming mono-calcium phosphate, and this in turn is acted on by the calcium carbonate forming dicalcium phosphate. The results may be summed up as follows: (1) The larger the quantity of calcium carbonate in the earth the more rapid is the retrogression of the superphosphate. The action is brought about by water. (2) The more water circulates in the earth the smaller is the retrogression and *vice versa*. The difference decreases, however, in proportion as the soluble phosphoric acid

phosphate is more satisfactory in clayey than in calcareous earths. The retrogression of the coarser superphosphate was again found to be slower than that of the finer. Further experiments showed that it would be better, if practicable, to add the manure after the germination of the seeds than before. A regulated amount of moisture is also seen to be of importance to the plants. The author finally concludes from a repetition of three experiments (1) That the coarser superphosphate acts more beneficially than the finer, and (2) that a larger



amount of moisture increases the action of the manure, both as regards the crop of seeds and of straw. *Experiments in the Field*: Similar experiments were made in the field with coarse and fine grained superphosphate (23mm. and less than 1mm. diameter respectively), with Kladnoer phosphate, precipitated calcium phosphate, ground bones containing 3 per cent. of nitrogen, and with sodium nitrate. The plants employed were barley, clover, broad beans, Tartary buckwheat, Hopetown oats, lentils, rye, etc. The manure was placed 7 to 13cm. deep in the furrows, covered with earth, and the seed placed above (3 to 8cm. deep). In these experiments the action of the coarse was better than that of the fine grained superphosphate. The Kladnoer phosphate in three cases produced a heavier crop, but was less satisfactory than the superphosphate in the case of potatoes. The precipitated calcium phosphate increased the crop, but much less so than the other phosphates. The bone meal was very beneficial on account of its nitrogen. The sodium nitrate only increased the weight of the beanstalks.—S. Y.

*On the Influence of Rain, Dew, and Artificial Watering on Plants.* Prof. Jul. Wiessner. Biedermann's Central-blatt. für Agrikultur-Chemie, 12, 471. Die Weinlaube, 1883, 9, 103-104.

THE author calls attention to the fact that when a plant is moistened the transpiration of its leaves is increased, and the flow of sap becomes more rapid. A leaf which has been moistened parts with water more rapidly than one which has remained dry. The increased transpiration is beneficial or the reverse, according to the moistness of the ground. If the earth is moist the plant receives more salts, if not it withers and may even die. The foliage of a plant should therefore only be watered when the ground is sufficiently moist to supply the increased demand of the roots. The action of the dew takes place after its evaporation, and is only injurious when the ground is very dry. Rain is generally more beneficial, since the earth is moistened at the same time as the foliage.—S. Y.

*On the Injurious Action of Ammonium Thiocyanate (Sulphocyanide) on Plant Growth.* Prof. M. Märker. Biedermann's Central-blatt. für Agrikultur-Chemie, 12, 497. (Report on the Efficiency of the Research Station for Agricultural Chemistry, Halle. Zeitschrift des landw. Central-Vereins f. d. Prov. Sachsen, 1883, No. 3, 74).

THE author found that an addition of 1 per cent. of this salt to superphosphate was not injurious. Oats were uninjured when treated with even 100 kilos. per hectare. In the English gasworks the liberated ammonia is utilised by passing it over superphosphate, by which it is absorbed with formation of ammonium thiocyanate (sulphocyanide). The author thinks that the prejudice against this superphosphate should be in great part removed by his results. On the other hand, C. Schumann (Biedermann's Central-blatt 1882, Vol. 1, 332) found that meadow-land treated with about 200 kilos. of thiocyanate per hectare was very injuriously affected, while a superphosphate which contained 25 per cent. of the salt produced very bad effect on grass and potatoes. P. Wagner (Biedermann's Central-blatt. 1873, 336) noticed a similar bad effect of superphosphate containing thiocyanate in the case of barley.—S. Y.

*On the Amount of Oxalic Acid Contained in Potatoes and Malt.* Prof. M. Siewert. Biedermann's Central-blatt. für Agrikultur-Chemie, 12, 499. Die Landw. Versuchsstationen, 28, 1882, 263, 270.

THE occurrence of an incrustation of calcium oxalate in the worm condenser employed for cooling the sweet mash in a large distillery, and the observation that the oxalic acid was present in the sweet mash, induced the author to determine the quantity of oxalic acid in the mash, and in the residues. (Schlempe.) The numbers obtained were as follows: In sweet mash, 0.155gm. per litre, or

0.059 per cent. when dry. In the nearly fermented mash, 0.155 gm. and 0.4 per cent. respectively. The oxalic acid in potatoes was obtained by boiling them, converting the starch into sugar with hydrochloric acid, precipitating the lime with sodium carbonate, and finally the oxalic acid with calcium chloride. After purifying the oxalate by repeated solution and reprecipitation, the oxalic acid was estimated, and found to amount to 0.017gm., or 0.0572 per cent. As this amount appeared too small to account for the whole of the oxalate in the sweet mash, further experiments were tried with barley, malt, and germinated malt. Barley contained no oxalic acid, malt 0.0015, and germinated malt 0.064 per cent. (Vegetable glue contains 1.59 to 17.43 per cent. of oxalic acid.) Thus in breweries, where only germinated malt is made use of, the deposition of calcium oxalate is small; but as green malt is usually employed in distilleries, a portion of the oxalic acid in the malt and residues is probably derived from this source.—S. Y.

*Study of the Development of Cherries and Currants.* Dr. Carl Amthor. Biedermann's Central-blatt. für Agrikulturchemie, 12, 499. Zeitschrift für Physiologische Chemie, vol. 7, 1883, 197-208.

FROM numerous determinations the author arrives at the following conclusions: The quantity of water and of solid residue in the case of cherries increases constantly during growth until the fruit is ripe. During the investigation this increase amounted to 398 per cent. of water and 295 per cent. of solid matter. The increase of the two is not always in the same ratio. Four weeks before maturity the increase of water is slower, before that time more rapid than that of the solid matter. The increase of ash and of phosphoric acid amounted to 204 and 195 per cent. respectively, but the ratio of these components to the water and solid residue decreases, the increase of the latter being more rapid. The percentage increase of ash and phosphoric acid in the stone amounted to 19.1 and 20 respectively. In the case of currants, both the water and solid matter increased during the investigation, which extended over six weeks, by 62 per cent. The ash during the same period increased by 50, the phosphoric acid by 47, and the sulphuric acid by 126 per cent.—S. Y.

*The Influence of Extreme Cultivation of the Soil on the Nutritiveness of Straw.* Prof. Märker. Biedermann's Central-blatt. für Agrikulturchemie 12, 500. Zeitschrift für Physiologische Chemie, vol. 7, 1883, 76-77.

THE influence of extreme cultivation appears to be prejudicial to the quality of the straw. In carefully-cultivated soil in the province of Saxony, the percentage of protein in oat-straw was found during three years to amount frequently to only 1 per cent., instead of the normal mean amount given by Wolff as 4 per cent. The injurious effect of over-cultivation may, however, be obviated by the employment of large quantities of manure. This result was brought about experimentally with Chili saltpetre and superphosphate.—S. Y.

*Experiments with Potassium Manures.* Prof. Fr. Farsky. Biedermann's Central-blatt. für Agrikulturchemie, 12, 457. Bericht der landw.-chemischen Versuchsstation in Tabor für das Jahr 1881 und 1882.

IN completion of earlier researches on the action of potassium manures (this journal Jan. 11, 1882, 210), the following experiments were undertaken—*Experiments with Pure Potassium Salts*: The chief manure employed was finely-ground bones, which was spread in the furrows beneath the seeds, and afterwards covered with earth. The potassium salt was either placed similarly under the seeds, or it was thrown three days later over the seeds, or lastly, it was placed regularly alongside the rows of plants. The results of the experiments showed that the crop was always decreased by the employment of potassium salts, the action varying with the position of the salt. The diminution was greatest when the salt was placed directly under the seeds, but less when the depth of the manure below the seeds was

greater, and much less when the salt was placed alongside the rows of plants, and therefore at some distance from the plants. The action of the chloride was less injurious than the sulphate. The after-effect of potassium salts was beneficial in the case of barley and buckwheat, the chloride being preferable to the sulphate, but the other plants could not stand the quantity of the salt employed. *Experiments with Stassfurt Manures*: (The composition of these manures is given in the above journal, Jan. 11, 1882, 490.) Similar results were obtained with these manures in the case of Tartary buckwheat and rye. Their influence was injurious, but the more dilute the solution in the earth the less marked was the action. From experiments with numerous other plants, the following conclusions were drawn: (1) The Stassfurt manures acted injuriously on barley, once also on oats, and to some extent on rye and clover, but beneficially in the other numerous cases. (2) The manure should be added in the autumn rather than the spring. (3) In the great majority of cases the chloride was more beneficial than the sulphate. The after-effect of the manures was generally beneficial in the few cases noticed. S. Y.

*On the Reduction of Nitrates in Arable Land.* Déherain and Maguenne. Biedermann's Central-blatt. für Agrikulturchemie 12, 506. Annales Agronomiques 1883, vol. 9, No. 1, 6-21.

It is well known that meadow land is much richer in nitrogen than tilled land. The researches of Déherain at Grignon showed that a soil on which maize or corn was grown became considerably poorer in nitrogen, while an unmanured soil planted with Esparto grass became gradually richer (*Central-blatt.* 12, 145). The researches of the authors on the reduction of saltpetre by ferments have been already described (*Central-blatt.* 12, 82). The difficulty of explaining the increasing richness in nitrogen of soil unexposed to the oxidising action of the atmosphere was rendered greater by Schlösing's observation that in such soils the nitrates disappeared through the nitrogen being liberated in the gaseous state. This is now better understood, and such a loss is but little to be feared, since the denitrating butyric acid ferment can only be formed in complete absence of oxygen; and in every analysis of the air contained in soil oxygen has been found to be present. In very dense soils the air might be completely removed by heavy rain, and the observation by Boussingault of the absence of nitrates in certain soils after continued rain may perhaps be thus explained. It is possible that in tilled soil there might be spots where the air could not penetrate, or where the air in the soil was completely robbed of oxygen, and where the ferment could then be developed; but the influence of the ferment could not be very great on account of the corresponding retardation of the nitrification of the soil. The comparison by Lawes, Gilbert, and Warington of the nitrogen present in drain water as nitric acid with that contained in the crops, and the researches of the authors at Grignon on the loss of nitrogen by different methods of cultivation, allow of a more correct appreciation of the injurious effect of rapid nitrification. It appears that by retarding the formation of saltpetre, the consequent loss of nitrogen is decreased, while Schlösing's observations point to the fact that in a soil exposed to an atmosphere poor in oxygen the formation of saltpetre is slow, and there is no doubt that an excess of carbonaceous matter reduces the oxidising power of the air. Thus, one of the causes of the richness in nitrogen of unworked land is the slowness of nitrification. The great quantity of carbonaceous substances present in untilled soils affords an explanation of their increasing richness in nitrogen. Only two causes of such increase are at present known—(1) The fixation of nitrogen observed by Berthelot, and (2) the fixation of atmospheric ammonia noticed by Schlösing. Whether one or both of these causes are at work, the necessary condition is the presence of humus derived from the residues of previous plant growth, and it has been found by Boussingault, Truchot, and other observers that meadow land is much richer in humus than ploughed land. In such soils, then, the fixation of nitrogen from the atmos-

phere is possible, while the loss of nitrogen is small, because the air penetrates with difficulty into the soil, so that nitrification is retarded. At the same time the reduction of nitrate by the butyric acid ferment cannot be great, for there must be enough oxygen present in the soil to allow of free respiration by the roots.—S. Y.

*On the Great Value of Nitrogen in Bone Dust.* Prof. Wagner Biedermann's Central-blatt. für Agrikulturchemie 12, 563. Zeitschrift für die landwirtschaftlichen Vereine des Grossherzogtums Hessen 1883, No. 12, 92.

THE bone dust of commerce contains from 1 to 4 per cent. of nitrogen, the mean being about 3.2, and 20 per cent. of phosphoric acid. The variation in the quantity of nitrogen depends on the method by which fats have been extracted. By boiling, steaming, or even drying nitrogen is lost, but if the fats are extracted by means of a solvent, such as benzene, this is not the case. Analyses of bone dust prepared in this way show a high percentage—4½ to 5—of nitrogen, and this method is therefore to be recommended.—S. Y.

*On the Manuring of Moorland.* H. J. Carsten-Hoogveen. Biedermann's Central-blatt. für Agrikulturchemie 12, 261 Landbouw-Courant, 32 Jahrg., 1882, No. 3, 10.

EXPERIMENTS were made to ascertain whether fields treated with lime in 1879 yielded in 1880 better crops than those without lime, but otherwise similarly manured with Peru guano. The lime was apparently without influence on the crop of oats. Some fresh land was manured for five consecutive years exclusively with Peru guano, 600 kilos per hectare the first year, and 450 kilos afterwards, with the result that the crop—black oats—increased gradually from 36 hectolitres the first year to 55 in the fifth. It thus appears that moorland may be converted into productive arable land solely by the use of artificial manures.—S. Y.

*Crushed Cotton Seed from Undecorticated Seeds.* Dr. Dietrich. Deutsch. Landw. Presse. Biedermann's Central-blatt. Agrik. Chem. 6, 428.

THIS product possesses a darker colour than that prepared from the decorticated seeds and is less expensive. Two samples analysed gave the following results:—

	I.	II.
Protein .....	23.56	23.06
Fat .....	6.26	7.01
Extractive matter free from nitrogen	24.94	27.12
Cellulose .....	25.73	25.53
Mineral matter .....	8.15	6.39
Water .....	11.36	10.69

E. G. C.

*On the Estimation of the Value of Different Samples of Bran.* Dr. J. Nessler. (Wochenbl. Landw. Vereins in Baden, 1883, 60). Biedermann's Cent.-blatt. Agrik. Chem. 6, 428.

THE generally-received idea that fine bran is better than coarse is not correct. In purchasing bran it is much more advisable to examine whether the sample contains dark brown or black fragments (seeds of tares), or long thin pieces of chaff or straw. In both these cases the bran is less valuable.—E. G. C.

*Tobacco Manures.* Dr. J. Nessler. (Wochenbl. des Landwirtschaftl. Ver. in Baden, 19, 152-154). Biedermann's Cent.-blatt. Agrik. Chem., 9, 642.

TOBACCO requires for vigorous growth a considerable amount of potassium. This should not, however, be applied in combination with chlorine, nor should other chlorides, such as common salt, calcium chloride, etc., be made use of, as a badly-burning tobacco will be the result. Potassium nitrate and sulphate, on the other hand, increase the combustibility of tobacco manured with them. Stable manures and the like, which are generally rich in chlorides, should be used with caution,



Comparatively little phosphoric acid is required, but nitrogen must be present in considerable quantity, and in an easily assimilable form. Tobacco needs much moisture during its rapid growth, and a light moist soil agrees with it best.—E. G. C.

*On an almost entirely Barren Soil from California.* Dr. A. Voelcker. Journ. Royal Agric. Soc., 2 ser., vol. 19, 236-237.

This soil possessed the following composition:—

Soluble organic matter . . . . .	with 0.99 nitrogen	0.75
Insoluble organic matter . . . . .		2.44
Oxide of iron . . . . .		4.48
Alumina . . . . .		4.54
Carbonate of lime . . . . .		2.58
Magnesia . . . . .		1.50
Potassium nitrate . . . . .		0.17
Sodium chloride . . . . .		0.04
Sodium . . . . .		0.58
Potassium . . . . .		0.51
Sulphuric acid . . . . .		trace
Phosphoric acid . . . . .		0.19
Insoluble siliceous matter . . . . .		81.92
		100.00

The explanation of the unproductiveness of this soil is to be found in the circumstance that its reaction was strongly alkaline. It yielded to cold water  $\frac{1}{2}$  per cent. of mineral constituents, consisting of carbonates of soda and potash, sodium chloride, and sodium nitrate. In order to convert such a soil into fruitful land, the author knows of no other method than to inundate from time to time with water, and so wash out the excess of alkaline salts.—E. G. C.

*A Further Proof that the Albumen of Living Protoplasm possesses a Different Constitution from that of Dead Protoplasm.* (O. Loew. Arch. für Physiol. 1883, vols. 7 and 8, 348-362.) Biedermann's Cent.-blatt. Agrik. Chem. 9, 645.

THE blackening which living plant-cells exhibit under the influence of a highly dilute ammoniacal silver solution is, according to the author's previous researches, to be attributed to the peculiar chemical character of living albumen. No doubt the relatively large quantity of metallic silver which the living cells are able to separate shows that the principal substance of the cell-contents, the albumen, is concerned in the reaction. The author first of all isolated and analysed the albumen of the alga, *spirogyra dobia*, in the fresh condition, and then, from the same alga, after treatment with silver solution, he isolated a silver compound and also analysed this. On a comparison of the albumen of the fresh alga with that in the silver compound it was found that the latter was much richer in oxygen than the former. From the alga previously treated with alcohol a similar silver compound could not be obtained. In the existence of this compound, the author perceives a proof that only living albumen takes part in the blackening of the cells.—E. G. C.

*Analyses of Various Cattle Foods.* Dr. A. Voelcker. Journ. Royal Agric. Soc., 2 ser., vol. 19, 237-242.

GREEN rye, preserved in silos, had the following composition:—

Moisture . . . . .	72.50
Lactic acid . . . . .	0.80
Nitrogenous substances (with 0.31 N) . . . . .	1.99
Extractive matter . . . . .	12.86
Cellulose . . . . .	9.72
Ash . . . . .	2.13
	100.00

This fodder was willingly taken by sheep and cattle; cows fed on it gave abundance of milk.

#### LOCUST BEANS.

	Pods and Seeds.	Seeds.
Moisture . . . . .	11.73	14.11
Fat . . . . .	0.62	2.03
Albuminoids . . . . .	5.62	16.91
Sugar . . . . .	40.61	—
Cellulose . . . . .	5.27	8.81
Extractive matter . . . . .	30.99	51.50
Ash . . . . .	2.76	3.61
	100.00	100.00

The pods contain a large quantity of sugar, and therefore less nitrogenous constituents, while the seeds contain scarcely any sugar. The pods, although a useful fodder, should not be given in too large amounts, because of their exceedingly sweet taste and the small proportion of nitrogenous matter present. The seeds ground to powder furnish an easily digestible and nonriching fodder. Dari-seeds (a kind of sorghum) and millet contained the following constituents:—

	Dari-seeds.	Millet Crushed.	Millet Uncrushed.
Moisture . . . . .	12.55	12.85	13.19
Fat . . . . .	2.93	3.91	3.83
Nitrogenous substances . . . . .	10.31	11.25	9.56
Extractive matter . . . . .	70.43	69.25	57.06
Cellulose . . . . .	1.63	7.73	12.51
Ash . . . . .	2.15	4.01	3.85
	100.00	100.00	100.00

Both foods are comparatively poor in albuminoids, and it is therefore advisable to mix them with fodder richer in nitrogen, such as crushed beans, etc. Russian linseed cake of great purity contained:—

Moisture . . . . .	11.13
Fat . . . . .	10.73
Albuminoids . . . . .	36.62
Extractive matter . . . . .	28.22
Cellulose . . . . .	7.91
Ash . . . . .	5.39
	100.00

E. G. C.

*Experiments on the Digestibility of Wheat Bran and its Alteration by Various Methods of Preparation and Administration, and on the Digestibility of Dry and Moistened Meadow Hay.* G. Kühn, F. Gerver, M. Schmöger, A. Thomas, O. Kern, R. Struve, and O. Neubert. Die landw. Versuchs. Stationen, Jahrg. 1883, 29, 1-214. Biedermann's Central-blatt. für Agrikulturehemie 12, 459.

THE object of the experiments was to ascertain whether the supposed increased wholesomeness of bran and other fodders when soaked in hot water, boiled, or administered in the form of broth, either alone or mixed with other fodders, was due to their greater digestibility when so treated. That no increase of digestibility in the case of bran is produced by boiling or disintegration was shown by previous experiments. As the bran could only be given in conjunction with another fodder, meadow hay, the digestibility of the latter when dry and moistened was first ascertained, and then the digestibility of the mixture under different conditions. From their experiments the authors draw the following conclusions: (1) The simple moistening of the hay immediately before its administration, either with water or with bran prepared as broth, has no sensible influence on the digestibility of the fodder, so long as the quantity of water employed is less than the total amount required by the animals. (2) Even when the amount of cold water employed is greater, it has no influence on the digestibility of the mixed fodder or on that of the bran. (3) The softening of the bran with cold water by standing for twenty-four hours has no influence on the digestibility of the mixed fodder or of the bran when the water taken by the animal in the ordinary way is not reduced by more than 50 per cent.; but when the quantity of water thus taken is reduced to below about 25 per cent. of the normal amount, the amount of raw protein digested is reduced both in the case of the mixed fodder and of the bran. (4) By soaking the bran with boiling water and allowing it to stand for twenty-four hours, the digestibility of the mixed fodder as regards raw protein is reduced, and the higher the initial temperature and the longer the application of heat the greater is this reduction. (5) The separate administration of the bran as a drink (in conjunction with dry fodder), and the administration of similarly-prepared bran mixed with the hay, give sensibly identical results. (6) The reduction of the digestibility of the raw protein when the bran is treated with boiling water is due to the action of the heat on the albumen of the bran, and it is the protein in the bran and not in the hay that is affected. Although only two kinds of fodder, meadow

hay, and wheaten bran were employed in these experiments, the authors consider it probable that their conclusions may be extended to other fodders. The methods of preparation described, as well as—in other cases—steaming, disintegration, fermentation, or simple heating, have no beneficial effect on the digestibility of the fodder, but rather reduce that of the protein, and they are therefore not to be recommended, except in special cases, when the fodder is rendered more palatable by such treatment.—S. Y.

*The Influence of Superphosphate on the Quality of Crops.* Prof. Fr. Farsky. Biedermann's Central-blatt. für Agrikulturchemie 12, 511. Bericht der landw. chemischen Versuchsstation in Tabor in den Jahren, 1881 bis 1882.

In some experiments on the action of Chili saltpetre and superphosphate as manures the author observed considerable differences in the crops of buckwheat and in the lateness or earliness of maturity. This is shown by the subjoined table:—

Description of Manure.			Moisture of Soil.	No. of days before Maturity.	Weight of seeds in grms.	
Chili Saltpetre.	Coarse Superphosphate.	Fine Superphosphate.			25c.c.	100 grains.
Not manured	—	—	Regulated	110	11'8254	0'9425
Ditto	—	—	Larger quantity	115	11'7995	0'9412
Saltpetre	—	—	Regulated	120	11'8294	1'0274
Ditto	—	—	More	120	11'8243	1'0272
—	Low percentage	—	Regulated	100	11'9836	1'1178
—	Ditto	—	More	106	11'9908	1'1479
—	—	Low percentage	Regulated	100	11'9075	1'1459
—	—	Ditto	More	108	11'8940	1'1463
Saltpetre	Low percentage	—	Regulated	110	11'8786	1'1572
Ditto	Ditto	—	More	115	11'8742	1'1580
Ditto	—	Low percentage	Regulated	110	11'8706	1'1568
Ditto	—	Ditto	More	115	11'8702	1'1556
Ditto	High percentage	—	Regulated	108	11'8877	1'2074
Ditto	Ditto	—	More	110	11'8694	1'2075
Ditto	—	High percentage	Regulated	108	11'8509	1'2064
Ditto	—	—	More	110	11'8500	1'2062

The harvest of buckwheat was gathered as soon as any seeds fell when the plants were gently shaken, whether they were still growing or not. The author draws the following conclusions: (1) The superphosphate acts as a stimulant and hastens the time of maturity. (2) The superphosphate increases the absolute and relative weight of the seeds, so that the present demands of the corn trade may be satisfied by its employment. (3) The Chili saltpetre does not increase the weight of the seeds, but rather delays the harvest.—S. Y.

*The Slow Weathering of Crude Bone Dust.* Prof. J. König. Biedermann's Central-blatt. für Agrikulturchemie 12, 562. Allgemeine Zeitung für deutsche Land-und Forstwirte, 12 Jahrg., 1882, 90, 551.

A field treated in the spring with stable manure and steamed bone dust contained no remains of bones even in the same year, while in other fields, manured from two to four years before with crude bone dust and stable manure, splinters of bone as large or even larger than peas were found. From analyses of these splinters, it would appear that the splinters in crude bone dust may lie in the ground four years before they are completely weathered.—S. Y.

*Improvements in the Manufacture of Artificial Fertilisers.* T. W. B. Mumford, London. Eng. Pat. 3343, July 5, 1883.

"I TAKE finely-ground commercial phosphorite, containing either a high or low percentage of phosphoric acid, and after dissolving the same in sulphuric acid of from 117° to 120° Tw., I agitate the mixture in a

vessel with either hot or cold water, passing the thin muddy product through an iron and lead filter press, or centrifugal or other suitable machine. The filtered solution contains the phosphoric acid in a soluble state, and the solid residue consists of a small percentage of soluble and insoluble phosphoric acid, sulphate of lime, and silica, and is a valuable fertiliser for certain crops. The solution of soluble phosphoric acid I run into shallow leaden pans, which may be situated on the top of pyrites or other kilns, or places where waste heat is available; and I evaporate the solution at a low temperature, until the liquid becomes highly concentrated and of a syrupy consistency. To this concentrated liquid I add, by means of an ordinary manure-mixer, or in other convenient way, a quantity of finely-ground ordinary commercial phosphorite. I have found the proportions of 1 part by weight of phosphorite to 2 parts by weight of the liquid to give good results. To these combined materials I add sulphuric acid of, preferably, 150° Tw. I have found that 1 part by weight of sulphuric acid to 6 parts by weight of the materials gives good results. The resulting product is a mixture resembling

ordinary superphosphate of lime, which in a few days becomes dry and hard, and capable of being ground or disintegrated into a very finely-divided and dry powder, and which, under ordinary atmospheric influence, does not lose its condition, and retains the phosphoric acid in a soluble state."

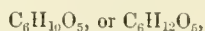
## XV.—SUGAR, GUMS, STARCHES, Etc.

*On the Formation of Mannite from Dextrose and Levulose.* F. W. Daferet. Berl. Ber. 17, 227.

C. SCHEIBLER has recently concluded from various observations that the formation of mannite from dextrose and levulose, by the action of sodium amalgam, is due to a secondary process (Berl. Ber. 16, 3010). The author has arrived at the same conclusion, at any rate as regards dextrose, from a study of the oxidation products of mannite. The products of oxidation of mannite vary according to the nature of the oxidising agent, the time of action, and the temperature: they are carbon dioxide, water, formic acid, mannitic acid, saccharic acid, inactive tartaric acid, and probably glycollic acid. Besides these a product is formed, when the oxidation is carefully conducted, which is identical with the mixture of a fermentable sugar and a substance resembling mannitan, first observed by Gorup-Besanez (Ann. Chem. Pharm. 118, 257). Further investigation of these bodies has shown (1) that the so-called mannitose belongs to the group of glucoses, and (2) that the manitose does not differ from grape sugar, as Gorup-Besanez states, only by its optical inactivity but also in its behaviour with



hydrochloric acid, sulphuric acid, Fehling's and Knapp's solutions. The similarity in the behaviour of mannitol and levulose is very striking, and the author would pronounce them to be identical if the former could be obtained in a clearly optically active state. The properties of the second body are not characteristic; it has an insipid taste, does not reduce Fehling's solution, is not fermentable, and is probably optically inactive, and thus resembles the little-known compounds mannitan and dextran. The author has never observed the formation of dextrose. When the mixture is treated with sodium amalgam mannite is re-formed, whence the author concludes that it cannot be in consequence of unsuitable conditions of oxidation that dextrose is not formed from the same mannite which is formed by the reduction of dextrose, when, under the same conditions, the easily decomposed mannitol is formed from mannite; or, in other words, that it is not from dextrose, but from a secondary product, possibly the second of the two substances mentioned above, to which Gorup-Besanez gives the formula—



that the mannite is formed by reduction. By renewed oxidation of the two substances the capability of reduction is diminished, whence they cannot be regarded as intermediate products between mannite and dextrose. The author has only recently been able to separate the mannitol from the other substance, and he is therefore as yet unable to say whether it is physically isomeric or identical with levulose, or whether it is also a secondary product.—S. Y.

*Record of a Year's Working of a Sugar Refinery.* G. Stade. Geitschrift des deutschen Vereins für Rübenzucker 1883, 797, and Dingl. Polyt. Journ. 251, 127, 1884.

A MINUTE and detailed account is here given of a year's working of a refinery which dealt principally with crude sugar from the district of Magdeburg and Brunswick. During that period 5777.3 tons of raw sugar passed into the factory, the average composition of this being—

		In 100 parts Dry Substance.	To 100 parts of Sugar.
Sugar .....	96.39	97.96	—
Water .....	1.60	..	—
Non-saccharine substances .....	2.01	2.04	2.09
Ash .....	0.99	1.01	1.03

The finished products were of six different qualities: the absolute yield of each, the amount obtained from 100 parts of crude sugar worked up, and the composition of each quality, are here given—

Name of Quality.	Absolute Weight Obtained	Amount of Pro- duct from 100 parts of Crude Sugar.	Composition per cent.			
			Sugar.	Non- Sugar.	Ash.	Water
Candy, white, & half white	Tons. 975.579	16.89	100	—	—	—
Candy, yellow and dark yellow .....	87.981	1.52	99.8	—	—	—
Lump sugar in loaves of 12½ kg. ....	1424.615	24.66	99.1 to 99.8	—	0.3 to 0.1	—
Lump sugar crushed .....	1591.965	27.55	99.4 to 99.8	—	0.3 to 0.1	—
Lump sugar in tablets .....	1021.250	17.68	99.4 to 99.8	—	0.3 to 0.1	—
Brown sugar..	118.667	2.06	91.37	5.71	2.36	2.72
Osmose-water concentrated	81.000	1.40	12.13	41.39	18.81	16.48
Syrup .....	316.230	5.47	56.61	26.65	9.79	16.74
Total .....	5617.307	97.23	—	—	—	—

It is thus apparent that a total loss of 159.993 tons took place—that is to say, 2.77 per cent. calculated upon the crude sugar. Now, from the known percentage composition of the raw sugar treated, it was calculated that 5568.7 tons of pure sugar passed through the works. Of this, 5424.3 tons were obtained as pure sugar in the finished products. The loss of pure sugar was therefore 144.4 tons—that is to say, 2.59 in 100 parts of pure sugar, or 2.50 in 100 parts of raw sugar. It was found that for every 100 parts of loss 19.33 took place in the animal charcoal, 25.92 in the sweet waste water, and 54.15 which could not be referred to any particular stage. Of the mineral matters a loss of 8.862 tons took place, this item passing out of the factory in the processes of filtration. The following summary will give a clear idea of the degree of efficiency attained to in these works: 100 parts of pure sugar in the crude material yielded 93.366 of refined sugar, 0.638 sugar in the osmose water, 3.346 sugar in the syrup, and 2.650 of loss. If the sugar in the osmose water and that in the syrup be brought under the one heading of syrup, that item becomes 3.984. If, however, the osmose water be regarded as loss, then the yield for every 100 parts becomes—93.366 refined sugar, 3.346 syrup, and 3.288 loss. As regards the consumption of fuel, 100 parts of crude sugar required 144.7 of Westphalian anthracite, with 1.9 per cent. of water and 10.8 per cent. of ash. This extraordinary consumption is due to the poor calorific value of this fuel, and is to be partially ascribed, moreover, to the plant and machinery not being of the most advantageous form.

W. D. B.

*Improvements in Extracting Sugar from Molasses, and in Apparatus connected therewith.* J. H. Johnson, London. Communicated by J. E. Boivin and M. M. D. Loisean, Paris. Eng. Pat. 3093, June 21, 1883.

THIS invention "is concerned with the extraction of sugar from molasses." A "molasso-calcareous mixture is prepared with suitable proportions of lime and diluted molasses," and "a snerate of hydrocarbonate of lime is formed with the said mixture" by treatment of it by CO<sub>2</sub>. The "snerate of hydrocarbonate of lime" is then washed, and then decomposed by CO<sub>2</sub>, and finally "the mixture of carbonate of lime and saccharine liquid resulting from this operation is subjected to the usual treatment in order to obtain sugar therefrom."

*Improved Processes for Defecating or Clarifying Saccharine Liquors, which Invention also comprises a Compound for Use in such Processes.* H. H. Lake, London. Communicated by H. A. Hughes, Camden, New Jersey. Eng. Pat. 3397, July 9, 1883.

To separate from cane-juice or other similar saccharine liquor "all or the greater portion of the matters contained therein other than sugar and water," the author of this invention adds to such juice or liquor bisulphite of lime before subjecting it to the ordinary process of defecation. He then treats the defecated juice by a weak solution of sulphuric acid, and then injects steam into it to drive off all SO<sub>2</sub>.

## XVI.—BREWING, WINES, SPIRITS, Etc.

*The Pasteurisation of Beer.* M. Schwarz. Biedermann's Central-blatt. für Agrikulturchemie 12, 560. Allgemeine Brauer- u. Hopfenzeitung, Jahrg. 23, 1883, 23, 227-228.

A STATEMENT by Behrend that yeast is only partially killed by warming, and that it recovers after a time, led the author to make some experiments to ascertain the truth of this assertion. Solutions containing yeast were exposed to temperatures ranging from 10° to 55° C. and the percentage of dead cells counted. The number rose from 16.6 per cent. at 40°, to 100 per cent. at 55°, and the cells after being heated to the latter temperature, when placed in fresh malt extract and kept at a temperature of 5° to 7°, showed no signs of life even after 40 hours. In practice the bottled beer is placed in cold water, which

is then heated by the introduction of steam, or the bottles are placed in a steam bath. The great point is that every bottle shall be raised to the required temperature, but an error frequently occurs because the temperature of the water is observed and not that of the beer; while the want of uniformity in the quality and thickness of the bottles, and the unequal temperature of the water when heated by steam, also lead to mistakes. Experiments made by the author showed that the temperature of the beer in such cases is from 2° to 3° lower than that of the water, though the whole of the beer in each bottle attains an almost uniform temperature. It is thus seen that the water should be heated for a considerable time to 58°. Beer which has been imperfectly heated, as well as beer which contains healthy yeast-cells, remains clear in a cold place, but at the ordinary temperature they both become turbid, and are found to contain living yeast-cells, bacteria, moulds, albuminates, and some resinous matter from the hops. The author finds that the turbidity of beer which has been properly heated is due to a separation of gluten, and that such turbid beer, when heated to 50°, becomes clear again and will remain so for a considerable time.—

S. Y.

*Improvements in the Manufacture of Maltose and its Applications.* J. Imray, London. Communicated by L. Cuisinier, Paris. Eng. Pat. 3437, July 12, 1883.

"ACCORDING to this invention, starch has added to it water and from 5 to 10 per cent. of what may be termed 'infusion'—that is to say, the liquid extracted from germinating grain. The whole is thoroughly mixed in a vessel heated slowly by a steam jacket to about 80° C. during about one hour. The liquid mixture is then poured into a close vessel provided with an agitator, and heated for about half an hour till the pressure is about an atmosphere and a half. The liquid is then cooled by surfaces kept cool by circulation of water, to about 48° C., and then has added to it 5 to 10 per cent. of infusion, according as saccharification is desired to take place less or more rapidly. When saccharification has continued about an hour, the juice is filtered by a filter press through filter paper supported on fabric, and the liquid product may then be kept for several days without alteration at a temperature of 48° C. The liquid is concentrated in a vacuum pan to about 28°, and filtered through animal charcoal in filters heated and closed in the usual way." Methods are described of treating "potatoes, and roots of like kind," and also "cereals, such as maize," directly.

*Improvements in the Means for Promoting and Improving the Fermentation of Wine, Beer, and other Beverages.* F. Wirth, Frankfort-on-the-Main, Germany. Communicated by A. Reihlen, Stuttgart. Eng. Pat. 3321, July 4, 1883.

"THIS invention relates to the preparation and use of fibres for promoting fermentation in wine, beer, and other beverages, and is based upon the fact that not only do woody fibres and vegetable cellular substances in their minutest form become impregnated with fungoid growths when they are in contact with substances in a state of fermentation, but they may also be impregnated with the same growths deriving their germs direct from the air under favourable conditions. The fibres impregnated with the said fungoid growths may be used as ferment instead of yeast, but without the disadvantages attending the use of the same." One of these advantages is stated to be that yeast always contains "a varied quantity of fungoid growths, some of which are diseased." To obtain the prepared fibres which he proposes to use instead of yeast, the inventor pours "pasteurised beer over clean woody fibres or cellulose. At a suitable temperature for fermentation, the fungoid growths which develop in the fermentation of beer, deriving further germs from their contact with the air, are thus multiplied." "For the purpose of fermenting beer wort, it is necessary to select suitable vegetable substance as feeding grounds for the fungoid growths, and then to fix these

fungoid growths upon the material selected." The inventor's specification describes his methods of procedure, and the various advantages which he believes to attend the use of his "prepared fibres" instead of yeast, in considerable detail.

*Improvements in Treating Alcoholic Liquors with Electricity to Purify and Impart to them the Character of Age.* Communicated by A. C. Tichenor, Alameda, California, U.S.A., to W. E. Gedge, London. Eng. Pat. 3877, Aug. 9, 1883.

THIS invention consists in "removing fusel oils or other impurities from alcoholic liquors" by "passing through them an electric current by means of wires and electrodes, one of which" electrodes, namely, the negative one, "is provided with a removable envelope or casing," which may be removed when it has become "thickly coated with a slimy or viscid substance," and another instantly substituted for it. The said removable envelope may be of "Canton flannel, woollen cloth, or other fibrous textile material." When the liquor treated is whisky, it should be treated "in tubs or vats of about one barrel capacity," by the current from a dynamo machine "having a strength or power equal to a Daniell battery of 40 cells." A current of about one-third of this strength should be used for wines. In treating wines, the electrodes used should be metal plates. In treating whisky, they should be carbon plates.

*Improvements in Preserving Milk, Fruit, or other Organic Substances.* W. H. Thew, Waterloo, Lancashire. Eng. Pat. 3058, June 20, 1883.

THIS invention consists: "First, in subjecting the substance to be preserved, and which is contained in a tin or other suitable germ-proof vessel, to a temperature which is sufficient to kill the fully-developed organisms, and which varies in different cases between 150° and 200° Fahr., and keeping it at this temperature for a period of between one and two hours. Secondly, in allowing the heated vessel to cool to the mean temperature of dwelling-rooms. Thirdly, in repeating these alternate processes of heating and cooling at intervals of between twelve to twenty-four hours, for a number of times, depending upon the substance to be preserved, but which usually is not less than five times. The theory of the process is the following: The more fully-developed organisms which are the immediate cause of the fermentations and decompositions to be prevented are killed by the repeated and lengthened exposure to 150° or to 180° Fahr. In the times which elapse whilst the substance is cooling from this temperature to that of the air, or which elapse during the heating up of the substance from the ordinary temperature to 150° or to 180° Fahr., the germs yet unacted upon are exposed to temperatures most conducive to their development, and developing become amenable to the destructive action of temperatures of 150° and 180° Fahr."

## XVIII.—SANITARY CHEMISTRY, DISINFECTANTS.

*Improvements in the Purification of Sewage Waters and the like, and the Recovery of Valuable Products therefrom.* Dr. J. Boek, Puschkau, Germany. Eng. Pat. 3252, June 30, 1883.

By this invention there is first added to sewage, per cubic metre thereof, "250grms. of wood pulp, or paper fibre, 140grms. of chloride of iron, 100grms. of concentrated solution of chloride of magnesium, and 1,500grms. of lime in the form of milk of lime." The mixture is allowed to settle, and then the supernatant liquor is decanted off. The precipitate, which is said to "contain all the phosphoric acid as well as all the nitrogen" of the sewage from which it has been precipitated, is pressed and dried for use as manure. The filtrate is first "treated by an oxidising agent," preferably "manganate of an alkali, in the proportion of about 100grms. per cubic metre," and then by a quantity of sulphurous acid a little greater than the quantity necessary to form



normal sulphite with all the lime in it. There is thus formed "a very small proportion of bisulphite of lime, which to a very high degree possesses the property of keeping the water entirely unaltered for a long time."<sup>4</sup>

### XIX.—PAPER, PASTEBOARD, Etc.

*An Improved Process of Making a kind of Fibrous Lig-nous Cellulose suitable for Paper-making and for other Purposes to which Cellulose is applicable.* A. M. Clark, London. Communicated by R. Blitz, Paris. Eng. Pat. 3240, June 29, 1883.

This invention consists in digesting "any kind of soft wood or other fibrous vegetable material" for from 4 to 8 hours, under a pressure of 3 to 4 atmospheres, with water to which has been added, per 127 cubic feet of wood to be treated, "13 pounds of hydrosulphite of soda, 6½ pounds of caustic soda, and 15 grains of vanadate of ammonia, dissolved in 60 grains of hydrochloric acid."

*Improvements in the Preparation and Manufacture of a Compound from Vegetable Materials suitable for Electro Non-conductors and Insulators; applicable also to other useful purposes.* E. C. T. Blake, Brixton. Eng. Pat. 3127, June 23, 1883.

By this invention, "one-third by weight of ground waste cork, and two-thirds of dry wood pulp or waste fibrous materials (advantageously waste cocoa-nut fibre)," are to be "mixed with the colloid of fibrin of blood, or instead thereof with paste or other material (advantageously made of rye or rice flour)," and the mixture, "in a thick pulpy state," is to be "put into moulds and kept under a continuously increasing pressure until all the watery parts are removed." The "boards, blocks, or other unmoulded forms" so obtained are then to be thoroughly dried, and thereafter well soaked "in boiling linseed oil or other waterproofing material," and then baked at 212° F. They are then to be again soaked in linseed oil or the like, and then to be again baked. They are said to be then "perfectly waterproof, and diluted acid proof," and "in appearance similar to ebonite."

*Improvements in Boilers or Vessels Employed in the Treatment of Fibrous Materials for the Manufacture of Paper Pulp, or for other purposes.* J. T. M'Dougall, Manchester. Eng. Pat. 3257, June 30, 1883.

In the lead-lined boilers which have hitherto been employed for digesting wood and other fibrous materials in acid solutions "a considerable difficulty has hitherto been experienced in keeping the lead in its relative position with the casing when the boiler or vessel is submitted to high temperatures. The repeated action of heat upon lead causes it to continually increase in area, and thus gradually accumulate into ridges or creases, which soon break, and necessitate the relining of the boilers or vessels. Most metals, such as steel and iron, expand when heated, and return to their original volume on cooling. Lead does not possess this power of returning to its original area after expansion." By the present invention it is proposed to secure the lining of lead-lined boilers "to the casing of the boiler, in such manner that the creeping or sliding effect of the lead is localised into sufficiently small portions to make the lead lining much more durable than" hitherto. This is to be accomplished "by providing a sufficient number of suitable holes through the casing, and corresponding holes in the lead lining, and passing through both these holes bolts or fastenings, having a head or the like on the inside, and being provided on the outside with one or more nuts or other means for tightening or holding the lead lining and the outer casing firmly together, so as to hold the lead lining in position to the casing. These bolts or fastenings should by preference be fixed at distances not very far apart, say about from 9in. to 24in. in every direction, according to the thickness of the lead lining, and the temperature to which the vessel is required to be subjected."

### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

*Action of Sodium upon Camphor.* J. Kaehler and F. O. Spitzer, Monatsh. f. Chem. 4, 494.

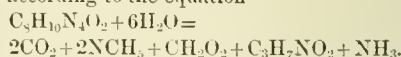
THE products of this reaction are, according to Baubigny, the sodium compounds of camphor and of borneol. The authors have investigated the reaction under the condition of exclusion of oxygen, viz., by employing a low-boiling petroleum ether as the solvent for the camphor, and allowing the reaction to proceed in an atmosphere of hydrogen. The sodium compounds were then dissolved in water, the solution filtered from a small residue of camphor, and decomposed by hydrochloric acid. The resulting precipitate was resolved, by fractional crystallisation into (a) white shining plates, melting point 140°, insoluble in water, freely soluble in alcohol and in ether, and having the composition  $C_{20}H_{30}O_3$ , and (b) camphoric anhydride,  $C_{10}H_{14}O_3$ . This paper is a preliminary notice of an extended investigation of the subject.—C. F. C.

*Papaverine.* G. Goldschmiedt, Monatsh. f. Chem. 4, 704.

PAPAVERINE yields, on fusion with potash, methylamine and dimethylhomopyrocatechol, together with protocatechuic acid, the latter being the chief product. It does not yield an acetyl derivative. Heated with hydrochloric acid at 130° it yields methyl chloride. The author is engaged in prosecuting the investigation, of which this is a preliminary notice.—C. F. C.

*Caffeine and Theobromine.* E. Schmidt, Dingl. Polyt. Journ. 251, 144, 1884. From Archiv. der Pharmacie, 221, 656, 1883.

ALTHOUGH caffeine is to be regarded as methyl-theobromine, or tri-methyl-xanthine, the author has not succeeded in converting it into theobromine by abstracting a  $CH_3$  group by the action of hydrochloric acid. He considers that hydrochloric acid induces the decomposition according to the equation—



For the preparation of theobromine, the author mixes cocoa of commerce with one-half of its weight of fresh slaked lime, and extracts the mixture with boiling alcohol of 80 per cent. On cooling the alcoholic extract, pure theobromine crystallises out. The base so obtained is a white crystalline powder in the anhydrous condition. It sublimes with little alteration at 290° C. and its analysis leads to the formula  $C_7H_8N_4O_2$ . To convert theobromine into caffeine, equivalent quantities of theobromine, methyl iodide, and potassium hydrate in alcoholic solution are heated for some time at 100° C. The theoretical amount of caffeine is not obtained, as a portion of the theobromine appears to suffer decomposition, but in its chemical and physical properties the caffeine thus produced appears identical with that occurring naturally, and with the product of the action of methyl iodide upon silver theobromine. The author has observed the presence of caffeine in the last mother-liquors, from which theobromine has been separated in its preparation, as above described, from cocoa.

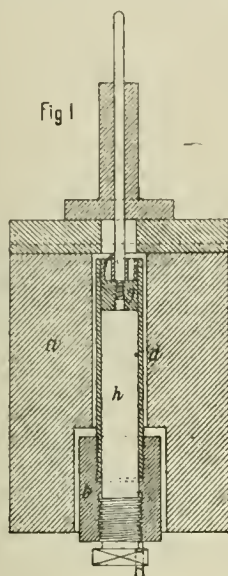
W. D. B.

### XXI.—EXPLOSIVES, MATCHES, Etc.

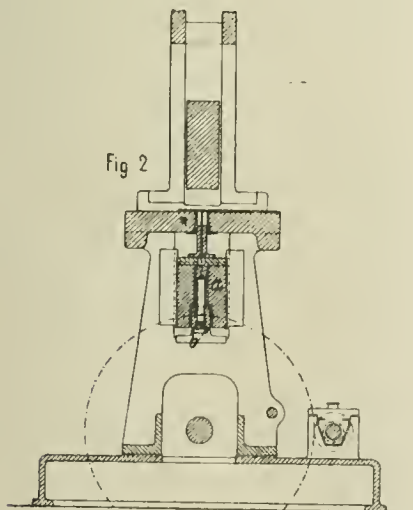
*Improvements in Explosives.* Dingl. Polyt. Journ. 251, 118, 1884.

SALANVILLE and Laligant (Ger. Pat. 19839, April 12, 1881) propose to employ a mixture of potassium or sodium hydrogen sulphate, potassium or sodium nitrate and glycerin, which is said to produce nitro-glycerin when ignited by means of a priming composed of potassium chlorate and carbonaceous matter. The explosion is stated to be direct. J. Gemperle (Ger. Pat. 23933, December 10, 1882) prepares "amidogene" as under: 73 parts of potassium nitrate and 1 part of magnesium

sulphate are dissolved in one-third of their weight of boiling water. On solution being complete, 8 parts of ground wood charcoal, 8 parts of bran, and 10 parts of sulphur are added, and the whole allowed to digest for 2 hours at  $140^{\circ}\text{C}$ . The mixture is dried for 5 hours at  $50^{\circ}\text{C}$ . and compressed into cartridges under a pressure of 5,000 atmospheres—a pressure which is ten times as great as could possibly be required, and which is likely

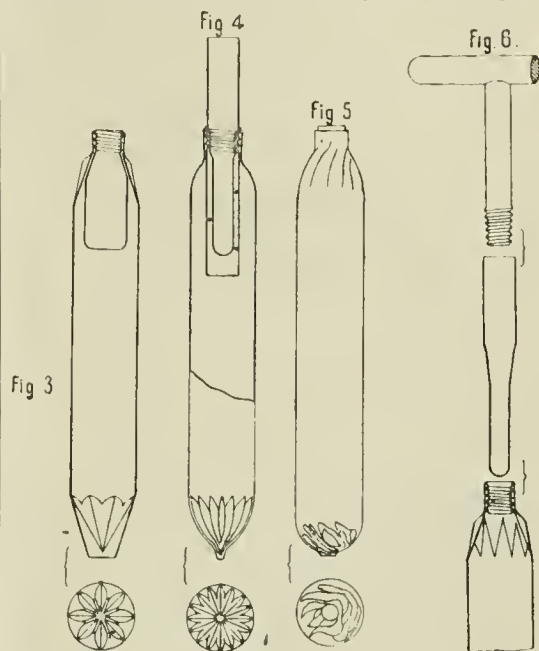


to give rise to accidents. It is claimed that the operation of compressing this compound is absolutely devoid of danger. Such a statement is to be received with caution, for although the elasticity of the bulky carbonaceous matter possibly renders that operation less dangerous than is the case with ordinary black powder, yet experience shows that no explosive can be very power-



fully compressed without risk. In order to decrease the length, and thereby increase the durability, of the needle required for the perforation of prismatic powder, Schwartzkopf (Ger. Pat. 16712, April 17, 1881) adopts the arrangement here illustrated. In the centre of the bearing guide *a* of the inferior plunger *b* a rectangular cavity is cut, its sides being parallel to the sides of the guide. At its lower end the plunger *b* is actuated. Into

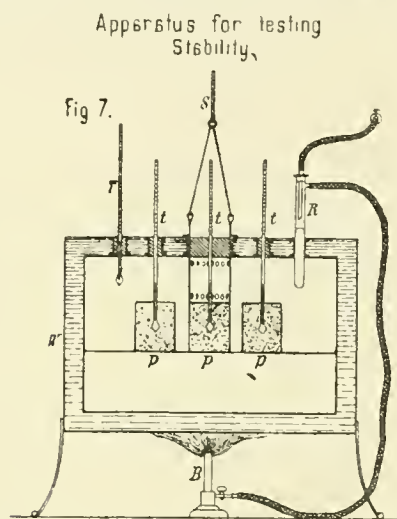
*b* the pin *h* is screwed, and *h* presses forward the cylinder *g*, to which the needle is attached. The cylinder *g* is retained by the cap *f*, which is kept in position by the tubes *d* screwed into the upper side of the plunger *b*. In the operation of compressing powder cake, Glaser (Ger. Pat. 16763, July 17, 1881) advocates the employment of ebonite press plates, which, by reason of their elasticity, oppose continuous resistance to the pressure without being injuriously affected as to the regularity and smoothness of their surface. The plan devised by Gruson and Hellhoff (Ger. Pat. 19430, March 3, 1882) for the application of liquid explosives is an attempt to solve a most difficult problem, one which, so far as nitroglycerin is concerned, yet remains unsolved. Tubes of tin plate, with soldered seam, are cut to the right length and closed at the bottom either by forcing the end through a stellate die, as shown in Fig. 3, by folding the



end in plaits by means of a tightened cord, as in Fig. 4, or by twisting up the end with a screwing motion, as in Fig. 5, and in each case soldering up the joints. In the neck an iron cap, screwed internally at its mouth, is soldered. This serves to contain the detonator, whose shell is made of tin, and is kept in position by being pressed into the female conical screw of the iron cap by means of the male screwed tap shown in Fig. 6. The detonating agent is placed in this tin cap, surrounded with the safety fuse, which is fixed by nipping the sides of the tin tube upon it. The whole detonator is then rendered impervious to water by means of cement. The patenting of a method for preparing quick-matches from straw haulms would rightly be considered a retrogressive step in these days of the *Bickford safety* and other fuses, were it not that in some localities the miners adhere most tenaciously to the old form. Pietzka (Ger. Pat. 16866, May 14, 1881) fixes the haulms gathered together in a bundle in a frame, and brings the ends into the conical mouthpiece of the same funnel through which the fine-grained powder is shaken into the haulms. In the *Mittheilungen über Gegenstände des Artillerie- und Genie- Wesens*, 1883, 85, Hess contributes his results of a series of experiments in connection with the exudation of nitro-glycerin from dynamite, consequent on the repeated freezing and thawing of the cartridge. A cartridge of kieselguhr dynamite, containing 71.63 per cent. of nitro-glycerin, was imbedded in kieselguhr, and frozen and thawed alternately three times. The surface was then found to contain only 67.52 per cent. of nitro-glycerin. The average amount of nitro-glycerin in the whole cartridge



had sunk to 71.42 per cent. The cartridge was then wrapt in paper saturated with paraffin, and again frozen and thawed three times. The percentage of nitro-glycerin at the surface was found to be 70.99. It is evident that in the first instance the loss was due to an absorption of the exuded nitro-glycerin by the surrounding kieselguhr. To test the stability and complete purification of explosive substances principally of the nitro-compound class, Hess proposes to expose the same in an air-bath to a temperature of 70°C., and compare the indications of a thermometer, whose bulb is plunged into the explosive, with those of one in the air-bath, it being assumed that the cause of an excess of temperature is incipient decomposition of the explosive. The apparatus here figured (Fig. 7) consists of the bath



W, provided with the thermo-regulator R and thermometer T. The explosive under examination is contained in the vessels P P P. These have double sides, the intervening space being filled with kieselguhr. Thermometers *t* pass through the top of the bath and rest in the explosive, of which samples of about 100grms. will suffice to afford reliable results. To obviate any possible danger during the testing of doubtful samples, the arrangement of the middle tube may be adopted. This is perforated near the top, closed with a cover, and suspended by a flange. By means of the cord S, which passes over a pulley, the explosive may be easily removed from the bath and plunged into water. The method appears simple, and possesses the advantage over the tests hitherto in use that a larger quantity of the explosive can be taken. As, however, dynamite, for instance, usually occurs in commerce in cartridges of less than 100grms. weight, it would be very interesting to observe whether the quantity of material taken for this test has any influence upon the results. For waterproofing the Bickford safety fuse, tallow may be employed, but Hess recommends a mixture of 6 parts of wax, 1 part of asphalt, and 1 part of resin, melted at 160°C. It is, however, useless to attempt to waterproof a Bickford fuse when the spark is sufficient to rupture the casing, for that would at once permit the ingress of water. With the aid of Sébert's Velocimeter, Sébert and Hugoniot confirm the fact, often hitherto observed in practice, that the velocity of explosion of a charge is directly proportional to the resistant pressure. The dynamite employed in the criminal attempts upon the Government and Times offices, on March 15th, 1883, was shown by Dupré to possess the following composition: Nitro-glycerin, 29.8 parts; sawdust, partially carbonised, 63.8 parts; moisture, 6.4 parts. The sawdust was coarse, and of a dark brown colour. The specific gravity of the dynamite was only 0.33. This dynamite was not very sensitive to percussion, and when detonated in a lead cylinder produced a cavity of 17 volumes as compared with that of 45 produced by Nobel's

No. 1 dynamite. The accidental explosion of five incorporating mills in the works at Basinghall, owing to lightning, demonstrates the necessity not only of providing danger buildings with sufficient lightning conductors, but likewise of keeping their earth terminals in thorough working order. The avoidance moreover of sharp bends in the conductor is very desirable. In the *Zeitschrift des österreichischen Ingenieur- und Architektenvereins*, 1882, page 121, Hajnat, Santay, and others publish interesting results of the methods employed in quarrying limestone for the construction of the harbour of Fiume. The three principal quarries worked were those of Martinschizza, Zurkovo, and Prelucca. In the two former the limestone was full of interstices, in the latter only was it compact. By reason of its price and convenient form, the explosive known as "diorexine" was exclusively employed. Diorexine is composed of 42.78 parts of potassium nitrate, 23.16 parts of sodium nitrate, 13.40 parts of sulphur, 7.49 parts of wood charcoal, 10.97 parts of beech-wood sawdust, 1.65 parts of picric acid, 0.55 parts of water. The following figures as to the quantity of explosive required for 1 cubic metre of stone show that in compact rock, chamber mines, in which the quantity of explosive fired at one time is very large, are by far the most economical but in a non-compact formation the plan of bore-holes gives the best results. The quantity of diorexine required for 1 cubic metre of stone was:—

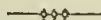
	With bore holes K.	With chamber mines. K.
In Prelucca (compact formation) ..	0.369	0.290
In Martinschizza (loose formation). 0.278	0.278	0.485
Zurkovo (loose formation) .....	0.199	0.522

For the production of the cavities for the smaller chamber-mines, advantage was taken of the action of hydrochloric acid upon the limestone. The acid was introduced into the bore-hole by means of a copper tube, and when a sufficiently large cavity had been eaten out, was withdrawn, and used repeatedly in new bore-holes until it was nearly neutralised. The plan was found to be very simple, but more tedious and expensive than the usual method of blasting out the required chamber with a small quantity of dynamite. In blasting submerged rock the expense of and time occupied in preparing bore-holes to receive the charges is no inconsiderable item of the whole undertaking. Lawer (*Mittheilungen über Gegenstände des Artillerie- und Genie-Wesens*, 1882, 1, and 1883, 437), taking advantage of the resistance offered to the explosive force by a considerable weight of water, has employed, with success, unconfined charges simply placed upon the surface of the rock. The blasting proceeded along the rock in a direction contrary to that of the current, so that the loosened masses were for the most part swept down the stream. An anchored barge, with projecting scaffold, was provided with a long beam or ram movable in the direction of its length upon the scaffold. Sockets were placed at equal distances upon the ram, and in any of the sockets could be fixed a long rod of iron tube suspended by a chain or wire rope. This rod carried the explosive at its lower end and could place it in almost any position, seeing that both the rod and the ram were movable, the one in a vertical, the other in a horizontal, direction. The chain or wire suspending the iron rod was passed over a pulley to a windlass, and so brought the rod completely under control. By this method of submarine blasting the consumption of explosive was greater than when the system of bore-holes was adopted, but the extra expense in this item was much more than counterbalanced by the saving in time and labour. From experiments recently made in America, interesting information as to the time elapsing between the explosion and its visible effect in blowing up a wreck has been obtained by photography. In 0.1 second after the explosion the vessel was broken up, and a column of water 20 metres high projected; in 1.5 seconds thereafter the column of water was 50 metres in height; in 2.3 seconds after the explosion shattered fragments and a column of water 60 metres in height were projected; in 3.3 seconds the water thrown up returned to the level, and after a total period of 4.3 seconds all was over.—W. D. B.

## New Books.

**FUEL AND WATER, WITH SPECIAL CHAPTERS ON HEAT AND STEAM BOILERS: A Manual for Users of Steam and Water.** From the German of Franz Schwaackhöfer, Professor at the Imperial and Royal School of Agriculture, Vienna. Edited by WALTER R. BROWNE, M.A., M.Inst.C.E., late Fellow of Trinity College, Cambridge. With numerous Illustrations. London: Chas. Griffin and Company, Exeter Street, Strand.

AN 8vo volume, bound in black cloth, with Editor's preface of 1½ pages, table of contents, introductory chapter by the Editor of 18½ pages, and subject matter filling 229 pages. An alphabetical index concludes the work. With the text are interspersed 85 woodcuts and numerous tables. The headings of the various chapters are as follows:—Part I. Fuel: Introductory Chapter on Heat and Combustion. Chap. i., Physical Characteristics of Heat (Temperature, Specific Heat, Conduction, Condensation, etc.); ii., Fuel (varieties); iii., The Firing Arrangements: The Furnace, The Flues, The Chimney, Useful Effect of Furnaces (with Examples of Calculations); iv., The Boiler: General Construction of Boilers, Choice of Steam Boilers, The Simple Cylindrical Boiler, The Compound Cylindrical Boiler, The Internally-fired Boiler, The Fire-tube Boiler, The Ten-brink Boiler, The Water-tube Boiler, Safety Fittings to Boilers, Boiler Explosions, Feed-water Heaters, Steam Pipes. Part II. Water: Chap. i., Physical Properties of Water; ii., Composition of Natural Waters; iii., Influence of the Composition of Water on its Application; iv., Purification of Water; v., Production of Cold and of Ice.



ADDENDUM.—Woodcuts illustrating the apparatus referred to in the article "On the Examination of Petroleum," pages 161 and 162, will appear in the April number of the Journal.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1881.

3542 J. Clark, Ph.D., Glasgow. Improvements for preventing the corrosion of iron articles by acids, salts, moisture, gases, or vapours. February 19  
3553 H. McGillivray, Clayton. Apparatus for diffusing gases through liquids used in depriving them of their hydrocarbons. February 19  
3558 J. Clark, Ph.D., Glasgow. Improvements in extracting nickel from its ores. February 19  
3599 A. J. Boulton, London. Improvements in machines for reducing ores. Communicated by G. Raymond and A. Raymond, Chicago, U.S.A. Complete specification. Feb. 19  
3615 R. Baynes, Liverpool, and A. Bigland, Liverpool. Improvements in treating, refining, or bleaching cotton-seed and other oils. February 20  
3612 P. M. Justice, London. Improvements in furnaces for calcining or burning cement. Communicated by Carl Dietzsch, Malstatt, Germany. February 20  
3644 M. A. F. Mennons, Paris. An improved compound for the prevention of incrustations and of galvanic action in steam boilers. Communicated by R. de Martino, Meta, Italy. February 20  
3656 J. B. Alliot, London. Improvements in treating liquid or other materials to concentrate and dry the same, and apparatus therefor. February 20  
3660 J. Buser, Marseilles. An improved non-conducting composition. Complete specification. February 20  
3673 C. Wigg, Liverpool, and J. Waller Pratt, Runcorn. Improvements in the manufacture of soda and chlorine. February 21  
3686 H. Gerhartz, Cologne, Germany. A new process of manufacturing liqueur out of milk. February 21  
3697 J. Miles, London. A new or improved method of treating certain materials for obtaining therefrom a powder for polishing or burnishing surfaces and for other purposes. February 21  
3723 W. Lloyd Wise, London. Improvements in the manufacture of salicylic acids. Communicated by the Chemische Fabrik Vormals, Hofmann and Schoetensack, Ludwigshafen, Germany. February 21  
3733 A. K. Huntington, London. A new or improved method and apparatus for separating mixed gases. February 21  
3736 W. A. Byrom, Wigan, and J. A. B. Bennett, King's Heath. Improvements in the method of taking off the volatile products from coal, shale, and other hydrocarbons during the process of distillation. February 22  
3760 I. Frankenburg, Salford. Improvements in the manufacture of waterproof cloth. February 22

3766 H. H. Hazard, London. The condensation and absorption of the products of combustion from coal or other gas or oil used either for lighting or heating purposes. February 22  
3780 Baron L. M. Dulfus, London. Improvements in the preparation of unflammable pulp for the manufacture of paper and for other purposes. Communicated by Julien Ponty, Brussels. February 22  
3786 Baron L. M. Dulfus, London. Improvements in means for rendering objects incombustible. Communicated by Julien Ponty, Brussels. February 22  
3790 H. W. Kue Meyer, Varel-on-the-Jade, Germany. Improvements in the treatment of fibrous materials for the preservation thereof. February 22  
3798 A. M. Clark, London. An improved process of and apparatus for separating and purifying certain components of the washings or scourings of wool and product obtained therefrom. Communicated by A. M. Hall, New York, U.S.A. Complete specification. February 22  
3807 F. Pidduck, Heywood. An improvement in the preparation of black lead for polishing and preserving the surface of iron. February 23  
3808 F. Wirth, Frankfurt-on-Main, Germany. Improvements in the manufacture of artificial stones, especially artificial lithographic stones and other similar bodies. Communicated by L. Rosenthal, Frankfurt-on-Main. February 23  
3836 John and James Addie, Glasgow. A new process of oxidising sulphites and hyposulphites. February 23  
3839 G. Patchett, Stockton-on-Tees; J. Dixon, Skelton-in-Cleveland; and R. Teasdale, Darlington. Improvements in means or apparatus for the manufacture of bricks, paving blocks, or the like, from the slag or scoria of blast furnaces. February 23  
3842 F. Wirth, Frankfurt-on-Main. Improvements in treating malt husks for certain industrial purposes. Communicated by P. Ammann, Munich, Germany. February 23  
3843 W. T. Read, Kentish Town. The treatment and preservation of beer. February 23  
3848 A. M. Clark, London. An improved process of and apparatus for extracting juice and crystallised sugar from the refuse or bagasse of sugar-cane and from any other part containing saccharine matter. Communicated by T. Blossom Yale, Columbia, Texas, U.S.A. February 23  
3855 J. King, junior, Manchester. Improvements in apparatus for taking off the gas from gas retorts and delivering it into the hydraulic main. February 25  
3870 J. F. Belfield, Exeter. Washing and purifying coal gas. February 25  
3882 E. Capitaine, London. Improvements in the manufacture of starch. Communicated by Drumm and Company, Kaiserslautern, Germany. February 25  
3904 Leon Barbier, Paris. Improvements in the manufacture of glucose. Complete specification. February 26  
3963 G. W. von Nawrocki, Berlin. A method of and apparatus for distilling ammoniacal fluids in a continuous manner. Communicated by Dr. P. Seidler, Elberfeld, Germany. February 26  
3987 W. T. Walker, Highgate. Improvements in condensing apparatus for purifying coal gas. February 26  
3995 J. Sandeman, Glasgow. Improvements in oleaginous compounds for preparing or treating textile materials or fabrics of cotton or other vegetable fibres for receiving or retaining colouring matters, or other purposes. February 27  
3996 F. Groom Redman, Peterborough, and J. Butt, Newark. Making sewage refuse into artificial manure. February 27  
4002 R. Hannan, Glasgow. Improvements in explosives for firearms, for blasting, and for similar purposes. February 27  
4003 L. A. Brode, Glasgow. The rendering of linen or calico impervious to water and heat. February 27  
4028 W. J. Chrystal, Glasgow. Improvements in the manufacture of bichromate of soda. February 27  
4065 Ivan Levinstein, Manchester. A new or improved method of preparing nitro-aniline and its homologues and nitronaphthylamines. February 28  
4102 S. Pitt, Sutton. Improvements in the manufacture of bichromate of potassa and other chromates. Communicated by H. Pemberton, Philadelphia, U.S.A. February 28  
4115 H. Whitehead and R. Hodgson, Ettiley Heath, Cheshire. A new and improved method of and automatic apparatus for making common and fishery salt from brine by steam. February 29  
4122 J. McCulloch, Airdrie, N.B., and T. Reid, Glasgow. Improvements in coke ovens or retorts, and in apparatus connected therewith. Complete specification. February 29  
4127 H. H. Waddington, Manchester. Improvements in waterproof fabrics. Complete specification. February 29  
4178 J. Ballantyne Hannay, Cove Castle. Improvements in means or apparatus for removing lead from water. March 1  
4182 E. T. Pemberton, Liverpool. Improvements in brewing or producing ale and porter. March 1  
4195 and 4198 C. S. Gorman, Irvine. Improvements in the manufacture of bichromate of soda. March 1  
4201 A. Fiegel, Berlin. Management of fibre stuffs by a solution of sulphurous acid, which is mixed with muriatic or acetic acid, in order to the action. March 1  
4202 J. Foulis, Mussulburgh, N.B. Improvements in the methods or process of and apparatus for draining, concentrating, clarifying, and solidifying or consolidating substances and liquids, especially applicable for filtering paper-mill sludge and consolidating the substances contained therein. March 1  
4207 R. H. Hepburn, Westminster. Improvements in the means and apparatus for supplying steam and air, or air alone, to furnaces to cause more perfect combustion of fuel. March 1  
4244 F. W. Pittuck, Hebburn, and J. M. Hucklebridge, Jarrow. An improved method of purifying metallic copper in the presence of arsenic or antimony, or both. March 3



- 1268 S. Gilchrist Thomas, Westminster. Improvements in the manufacture of alkalis and alkaline salts. March 3
- 1273 Shirley Bowden, Holborn. A new or improved method of producing biphosphates of a high percentage from the slag resulting from dephosphorising iron by the basic process and from natural phosphates. Communicated by R. Schliwa, Waldeck, Germany. March 3
- 1295 H. H. Lake, London. Improved means for generating steam by chemical agents. Communicated by H. Grünberg and E. Hardt, Cologne. Complete specification. March 3
- 1298 A. J. Boulton, London. Improvements in regenerative coke furnaces. Communicated by the Schlesiische Kohlen und Cokeswerke, Gottesberg, Germany. March 3
- 1304 A. M. Clark, London. Improved apparatus for agitating the liquid in liming vats. Communicated by J. B. McEnally, Clearfield, Pennsylvania, U.S.A. March 3
- 1312 J. Walker Newall, Ongar. Improvements in regenerative furnaces. March 4
- 1313 J. Walker Newall, Ongar. Improvements in kilns for burning bricks and other earthenware goods. March 4
- 1325 R. A. Mossman, and J. Mayel Mayelston, Elloughton, Yorks. Improvements in the manufacture or preparation of aerated liquids of all kinds, and of the bicarbonates of potash, soda, and ammonia, and likewise of the sesquicarbonates of potash and soda. March 4
- 1333 J. Miles, London. A new or improved method of extracting metals from the material found combined with coal, commonly known as ironstone. March 4
- 1348 H. J. Haddon, Westminster. Improvements in anti-fouling, anti-corrosive, and preservative paints. Communicated by W. Denison Folger, Calcutta. Complete specification. March 4
- 1356 Dr. T. Weyl, Berlin. Improvements in the production of caseine peptone and peptonised milk. March 4
- 1361 A. D. Cohen and H. J. Emanuel, London. Improvements in the employment of substances and in the means of producing cold, which is also applicable for other purposes. March 4
- 1362 F. Wirth, Frankfurt-on-the-Main, Germany. Improvements in the manufacture of cream of tartar. Communicated by F. Dietrich, Munich, Germany. March 4
- 1392 A. Adair, Egremont, Cumberland. Improvements in the manufacture of phosphates and phosphoric acid. March 5
- 1397 R. Howarth, Wolverhampton. Improvements in apparatus for condensing steam or other vapours. March 5
- 1403 T. Welton, London. Improvements in deodorising faecal matter in water-closets, urinals, and similar places. March 5
- 1410 J. C. Mewburn, London. Improvements in dyeing and otherwise operating upon fibrous materials when in the form of slivers, skeins, or warps, and in machinery employed therein. Communicated by J. Stoltz, Roubaix, Nord, France. March 5
- 1415 G. W. Von Nawrocki, Berlin. A method of manufacturing new pigments. Communicated by P. Bottiger, Lodz, Russia. March 5
- 1417 W. S. Richardson and W. J. Grey, Gateshead. Improvements in the treatment of chloride of sodium for the production of bleaching powder and other materials therefrom. March 5
- 1452 G. C. Denison and J. E. Higgin, Northwich. Improvements in and relating to the manufacture of salt from brine and apparatus therefor. March 6
- 1480 W. L. Wise, London. The manufacture of mordants and colouring matters from glucosides, tannin, and bitter principles. Communicated by C. H. Knoop, Dresden. March 6
- 1482 J. H. Johnson, London. Improvements in cementation furnaces. Communicated by A. Berthomieu, Paris. March 6
- 1516 W. Scott, Glasgow. Improvements in the recovery of ammonia from waste gases. March 7
- 1563 H. A. Dufrene, Paris. Improvements in apparatus for cooling beer and other liquids. Communicated by N. Duboc-Barily, Sedan, France. March 7
- 1632 C. A. Caspersson, Forsbacka, Sweden. Improvements in the process of pouring and casting iron, steel, and other metals. Complete specification. March 10
- 1650 H. J. Haddon, London. Improvements in apparatus for treating textile materials with liquids or gases. Communicated by O. Obermaier, Lambrecht, Bavaria. March 10
- 1652 H. H. Lake, London. Improvements in kilns or furnaces for burning bricks, lime, and similar materials. Communicated by W. Eckardt, Dortmund, Germany. March 10
- 1663 C. H. Greville Williams, Hounslow. Process for the separation of benzene and its homologues from the liquid obtained by compression of petroleum gas. Complete specification. March 10
- 1679 B. Davy, Keighley, Yorkshire. An improved method for extracting grease from soap water. March 11
- 1684 L. Poensgen, Düsseldorf, Prussia. Improved process for unloading tin-plate refuse in hermetically-closed apparatus, and at the same time obtaining ammonia. Complete specification. March 11
- 1690 F. Hocking, Liverpool. Improvements in and relating to apparatus for condensing and cooling, particularly applicable for the production of fresh water from salt water. March 11
- 1698 W. H. Spence, London. An improved method of utilising the waste residues of fuel. Communicated by F. W. C. Waldeck, The Hague, Netherlands. March 11
- 1707 J. B. Thompson, New Cross, Kent. Improvements in bleaching. March 11
- 1708 A. M. Chambers, Thorncliffe, and T. Smith, Chapelton. Improvements in coke ovens, and in methods of and apparatus for collecting and utilising the products of combustion from such coke ovens. March 11
- 1711 Bruno Baron von Steinaecker, Lauban, Prussia. An improved manufacture of combustible gas for lighting and heating purposes, and in apparatus for this purpose, and for the application of such combustible gas to furnaces, ovens, kilns, steam boilers, and other industrial and domestic purposes. March 11
- 1716 A. W. L. Reddie, London. Improved evaporating or condensing apparatus. Communicated by E. Theisen, Lindenan, Germany. Complete specification. March 11
- 1718 W. Black, South Shields, and T. Larkin, East Jarrow. Improvements in furnaces for the manufacture of sulphate of soda or hydrochloric acid, or for roasting or calcining ores, or for analogous purposes. March 11
- 1726 C. D. Abel, London. An improved titration apparatus for alkalimetric and acidimetric determinations. Communicated by E. Greiner, Stützerbach, Germany. Complete specification. March 11
- 1761 W. Weldon, Burstow, Surrey. Improvements in the manufacture of bicarbonate of soda. Partly communicated by A. R. Pechiney, Salindres, France. March 12
- 1785 R. Lehmann, London. A new or improved process for the manufacture of hydrogen. Communicated by J. Oettli, Cossonay, Switzerland. March 12
- 1792 A. Southwell, Southampton. An improved composition or compound for covering boilers, pipes, ice safes, and other like bodies. March 12
- 1796 P. Layden and W. McLean, Jarrow-on-Tyne. Improvements in priming and anti-fouling compositions. March 12
- 1808 T. Lowe, Old Radford, Nottinghamshire. Boiler composition. March 13
- 1811 W. J. A. Donald, Glasgow. Improvements in the manufacture of chromates and bichromates. March 13
- 1825 W. P. Wilson, London. Improvements in the manufacture of gas for illuminating purposes, and in apparatus or means to be employed therein. March 13
- 1826 W. J. Gale, Clifton, Gloucestershire. Improvements in treating leather for the purpose of rendering the same water-repellant, water-proof, and more durable. March 13
- 1830 J. H. Johnson, London. Improvements in the preparation of halogen derivatives of tetra-methyl-diamido-benzophenone and analogous ketone bases. Communicated by H. Caro, Ludwigshafen-on-the-Rhine, Germany. March 13
- 1833 J. Parker, Kilmarnock, N.B. Improvements in stoves, or apparatus for drying or heating bricks or other articles. March 14
- 1865 R. J. Atcherley, Ph.D., London. An improved method and apparatus for the amalgamation of free gold. March 14
- 1879 J. Watson, Greenhithe, Kent. Improved apparatus to be used in the manufacture or preparation of cement. March 14
- 1902 W. R. Lake, London. Improvements in apparatus for drying and superheating steam, and for similar purposes. Communicated by L. Fouque, Paris. March 14
- 1929 C. S. Gorman, Irvine, N.B. Improvements in the manufacture of bichromate of soda. March 15
- 1936 J. Hodgkiss, Wednesbury, Staffordshire. Improvements in furnaces for melting metals by a new or improved process. March 15
- 1917 S. G. Thomas, Westminster and El Biar, Algeria. Improvements in the manufacture of phosphatic salts. March 15
- 1955 G. W. von Nawrocki, Berlin. A furnace for reducing zinc and other metals. Communicated by J. Quaglio, Frankfurt-on-Main, Germany. March 15
- 1961 C. D. Abel, London. The production of violet and blue colouring matters of the rosaniline series by treating aromatic amines with alkylised amido derivatives benzoylchloride and of benzophenone. Communicated by Farbwerke Vormal's Meister, Lucius, and Brüning, Höchst-on-the-Main, Germany. March 15
- 5002 C. W. Rees, London. Improvements in treating wooden articles so as to give them the appearance of metal, and compositions therefor. March 17
- 5021 F. Wirth, Frankfurt-on-the-Main. Improvements in colouring matters. Communicated by Prof. E. Erlenmeyer, Frankfurt-on-Main, Germany. March 17
- 5025 S. R. Smyth, London. Improvements in smelting iron ores, and in the apparatus employed therein. March 17
- 5038 J. H. Johnson, London. Improvements in the preparation of colouring matters suitable for dyeing and printing from halogen derivatives of tetra-methyl-diamido-benzophenone and analogous ketone bases. Communicated by H. Caro, Ludwigshafen-on-the-Rhine, Germany. March 18
- 5056 H. Brewer, junior, Manchester, and W. A. Brewer, Crumpsall. Improvements in and material for producing a deep black polish upon wood, leather, linocrusta-walton, metal work, and other surfaces. March 18
- 5070 R. Kennedy and W. Fairweather, Glasgow. Improvements in bleaching. March 18
- 5071 R. Kennedy and W. Fairweather, Glasgow. An improved method of oxidising sulphites and hyposulphites. March 18
- 5082 W. R. Lake, London. Improvements in machines for breaking or crushing stone, ore, and similar materials. Communicated by T. A. Blake, New Haven, U.S.A. March 18
- 5089 A. J. Boulton, London. Improved continuous malt-kiln. Communicated by H. A. Kolndorfer, Vienna, Austria. March 18
- 5096 F. Settle Barff, Kilburn. Improvements in the treatment of liquids and substances which are subject to fermentation. March 18
- 5118 H. T. Stinchcombe, Bedminster, Bristol. An improved and special composition paint for protecting ships' bottoms of every description and other submerged substances of a like nature. March 19
- 5119 W. Wyatt, Ellesmere. Improvements in the method of carrying out the process of softening and purification of water, and in apparatus for use therein. March 19

# THE JOURNAL OF THE Society of Chemical Industry.

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*Charles G. Crosswell, Palace Chambers, 9, Bridge Street, Westminster.*

## THE JOURNAL.

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## NOTICES.

In accordance with the provisions of the 9th by-law, notice is hereby given that those members whose names are placed in italics in the annexed list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Members are hereby invited to nominate fit and proper persons to fill the four vacancies thus created among the Ordinary Members of the Council, in accordance with the terms of the 9th, 11th, and 12th by-laws.

The bye-laws are inserted in the number of the Journal for July, 1883. They can also be obtained from the Secretary, who will supply the prescribed nomination forms upon application.

## ANNUAL GENERAL MEETING.

The next Annual General Meeting of the Members of the Society of Chemical Industry will be held at Newcastle-on-Tyne, on Wednesday, Thursday, and Friday, the 9th, 10th, and 11th of July next.

It is proposed that the morning of Wednesday, the 9th of July, shall be devoted to the transaction of the ordinary business of the Society, the election of office bearers, and an Address by the President; that on Wednesday afternoon the Members shall visit various Works; and that in the evening those of the Members attending the Meeting who choose to do so shall dine together. By the kind permission of Sir WILLIAM ARMSTRONG, K.C.B., the dinner will take place in the Banqueting Hall at Jesmond Dene. The dinner tickets will not include wine, for which separate arrangements will be made.

It is proposed that on Thursday, the 10th of July, the Members shall meet in the morning for the discussion of papers by Mr. W. S. SUTHERLAND, Mr. GEORGE BEILBY, Mr. JAMESON, and, it is hoped, Mr. HENRY SIMON, and perhaps others, on the treatment of coal for the obtainment therefrom of condensable volatile products; and that in the afternoon they shall visit various Works.

In the evening a *Conversazione* will be held.

It is proposed that Friday, the 12th of July, shall be entirely devoted to visits to various Works. It is intended that a special steamer shall convey the Members in the morning down the Tyne to the Work to be first visited, and that in the course of the afternoon the Members shall have an opportunity of proceeding by this steamer as far as Tynemouth pier, so that they may see the whole of the Tyne between Newcastle and the sea.

The meetings on the mornings of Wednesday and Thursday will take place in the Lecture Theatre of the Literary and Philosophical Society. The Wood Memorial Hall of the North of England Mining Institute will be available during the Meeting as a Reception and Writing Room. The Library of the Literary and Philosophical Society will also be available as a Writing Room.



Various Works in the district, in addition to those to which visits will be specially organised, will be open to Members during the Meeting on presentation of their cards of Membership.

Opportunities of seeing in operation the SIMON-CARVÉ process, and the JAMESON process, will be afforded during the Meeting.

Messrs. BAIRD & Co. have consented that any Members attending the Meeting who choose to proceed to Gartscherrie for the purpose, shall see the process at work there for obtaining ammonia from the gases from blast-furnaces; and Messrs. YOUNG and BEILBY have promised to show their process at two Works near Edinburgh.

Full details of the arrangements for the Meeting will be published in the June number of the Journal.

Numerous inquiries have been made for sets of the Journal for 1882. Some of the numbers for that year are out of print. Members who have not the volume for 1882, and wish to obtain it, are invited to signify that wish to the Secretary as early as possible. If a sufficient number of applications for that volume are received, the missing numbers of it will be reprinted.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 36, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

#### LIST OF MEMBERS ELECTED APRIL 24th, 1884.

Henry Aitken, Danoch, Falkirk, N.B., mining engineer.  
C. Torre Anderson, Messrs. Bass and Company, Burton-on-Trent, brewer.  
John Anderson, 133, Bath Street, Glasgow, analytical chemist.  
James M. Bennett, 12, Hamilton Drive, Hillhead, Glasgow, metallurgist.  
J. William Biggart, 29, Cathcart Street, Greenock, N.B., analytical chemist.  
Charles Blatherwick, Dunaivon, Helensburgh, N.B., Government inspector alkali works.  
G. H. Bostock, 9, Great John Street, Manchester, analytical chemist.  
Henry T. Bottinger, Elberfeld, Germany, director of the Farbenfabrik. (See form.)  
Charles Jul. Caspers, care of Messrs. Read, Holliday, and Sons, Huddersfield, commercial manager.  
Frederick Cheshire, Laboratory, Somerset House, London, W.C., assistant analyst.  
Saml. Crowder, Stafford Lodge, Anerley Park, London, S.E.  
Thomas Dawson, Stonecroft House, Milnsbridge, near Huddersfield, aniline dye manufacturer.  
Geo. Dyson, Sockburn, Darlington, chemical manufacturer.  
James Donald, 3, Queen's Terrace, Glasgow, manufacturing chemist.  
Thomas Felton, 217, Brook Road, Upper Clapton, London, E., manager oil distillery.

J. T. J. Findlay, Silvertown, London, E., Chemist.  
J. J. Finlay, 53, Renfield Street, Glasgow, analyst and assayer, Tharsis Company.  
Daniel Frazer, 113, Buchanan Street, Glasgow, pharmacist.  
A. R. Gillespie, 20, Lombard Road, Trinity, Edinburgh, director oil company.  
F. Hadkinson, Pamphila Oil and Soap Works, Mitylene, Mediterranean.  
G. J. Hamlen, Oakbank Oil Works, Midcaldor, N.B., chemist.  
Wm. Hawdon, care of Samuelson and Co., Newport Iron Works, Middlesbrough.  
Z. J. Heys, Stonehouse, Barrhead, N.B., calico printer.  
T. G. Hunter, 81, Thistle Grove, South Kensington, chemist.  
G. H. Hurst, 11, Cable Street, Salford, Manchester, analyst and lecturer.  
Alex. Johnstone, Chemical Laboratory, Blairlodge School, Polmont Station, N.B.  
Samuel Knowles, Stormer Lodge, Crumpsall, Manchester, calico printer.  
Nigel Macphie, Clippens Oil Works, by Johnstone, N.B. cashier.  
A. J. Higgin, Little Peter Street, Manchester.  
William Marshall, Eddlewood, Hamilton, N.B., ironworks chemist.  
William G. O'Beirne, 15, Campbell Street, Mary Hill, Glasgow, analyst.  
J. Edgar Poynter, Clydencuk, Uddingston, N.B., manufacturing chemist.  
H. A. Rademacher, Turn Lee Paper Mills, Charlestown, Glossop.  
R. Andrew Robertson, 42, Aytoun Road, Pollokshields, Glasgow, mechanical engineer.  
Albert E. Reed, Ely Paper Works, Cardiff.  
Adolphe Roques, 36, Rue Sainte Croix de la Bretonnerie, Paris, chemical manufacturer.  
Prof. Samuel P. Sadtler, F.C.S., University of Pennsylvania, Philadelphia, Pa., U.S.A.  
Walter Shaw, Sherdley Glass Works, St. Helens, glass manufacturer.  
Hubert C. Shorten, 8, Hetley Road, Uxbridge Road, London, W., student.  
R. Simpson, Grecian Terrace, Harrington, Cumberland, chemical student.  
George Smith, Ravensdene, Sutton, Surrey, manufacturing chemist.  
J. Stevenson, Broxburn, N.B., assistant chemist in oilworks.  
George Swinburne, 33, Frederick Street, Gray's Inn Road, London, W.E., gas engineer.  
G. Carruthers Thomson, 77, Hill Street, Garnet Hill, Glasgow, engineer.  
Jno. Thomson, Institution for Deaf and Dumb, Queen's Park, Glasgow.  
R. Tatlock Thomson, 138, Bath Street, Glasgow, analyst.  
W. Spencer Turner, 225, Oxford Street, Manchester, pharmacist.  
G. Watson, jun., 318, St. Vincent Street, Glasgow, manufacturing chemist.  
A. Wilson, 3, Victoria Place, Trinity, Edinburgh, manager.  
W. Wright, Fairfield Road Chemical Works, Droylsden, Manchester.  
Edwin Addenbrooke, 18, Cawley Road, South Hackney, assistant chemist Metropolitan Board of Works.  
H. J. Barnes, Phoenix Chemical Works, Hackney Wick, manufacturing chemist.  
W. H. Booth, Lonsdale, Flinton, near Manchester, consulting engineer.  
Thomas Dearden, 12, Heywood Street, Bury, Lancashire, technical chemist.  
J. R. Fletcher, Kersley Paper Mills, Stoneclough, Manchester, paper maker.  
W. E. Heys, Milton Terrace, Everton Road, Manchester, electrical engineer.  
J. M. Irving, 6, Booth Street, Piccadilly, Manchester, chemical merchant.  
John Paterson, Derwent Villa, Workington, Cumberland, mechanical engineer.  
Walter V. Robinson, 6, Market Buildings, Mincing Lane, London, E.C., chemical merchant.  
Ferd. Rohling, 65, Nelson Street, Oxford Road, Manchester, technical chemist.  
A. G. Salaman, Thornton House, Clapham Park, London, S.W., consulting chemist.  
C. H. Saul, 76, Mosley Street, Manchester, drysalter.  
Julius Schadt, 11, Charlotte Street, Manchester, agent for aniline company.  
J. Sellars, Fairfield, near Manchester, manufacturing chemist.  
T. R. Shaw, Mill Street Works, Pendleton, Lancashire, oil merchant and dyer.

#### CHANGES OF ADDRESS, ETC.

*A List of Changes of Address follows the List of New Members for the information of the Society's officers and others. The General Secretary trusts that members will second his efforts to make this List a complete and accurate record by notifying to him their changes of address as promptly as possible.*

Angus, John (late of Fenchurch Avenue), 47, Lime Street, London, E.C.  
Barnes, R. L. (late of Buckhurst Hill), Lancaster House Savoy, London, W.C.

Boucart, Robt. (late of Manchester), à la Boissière, près Genève, Suisse.

Brown, J. T. (late of King's Road), 3, Dartrey Road, Chelsea, London, S.W.

Clift, Jas. (late of Jervoise Street), Billhay Street, West Bromwich.

Cox, John (late of Mulheim), Hunstanton St. Edmonds, Norfolk.

Duggan, T. R. (late of South Hackney), 11, Alkham Road, Stoke Newington, N.

Gibbs, W. P. (late of Darlington), care of Messrs. Newton, Keats, and Company, Sutton, St. Helens.

Hulme, James (late of Macclesfield), Owens College, Manchester.

Irwin, J. T. (late of Blackburn), 177, York Street, Cheetham Hill, Manchester.

King, A. J. (late of Mining Lane), Ingersley Vale Bleach-works, Bollington, near Macclesfield.

Laws, J. P. (late of Highgate Hill), 3, Tyfield Road, Oxford.

Miller, John, 151, Glenpark Street (not Gunport Street as given), Glasgow.

Walker, H. W., 37, Fox Street, Greenock, N.B.

## London Section.

Chairman: David Howard.

### Committee:

Sir F. A. Abel.	R. E. R. Newlands.
H. E. Armstrong.	B. Redwood.
W. Crowder.	T. Royle.
C. Graham.	John Spiller.
S. Hall.	G. C. Trewby.
A. K. Huntington.	W. Weldon.
R. Messel.	J. Williams.

Local Sec.: Thos. Tyrer, Garden Wharf, Church Road, Battersea, W.

The remaining meetings of the session will be held as follows, when papers will be read by—

On May 5th, Dr. Percy Frankland, "On the Composition and Illuminating Power of Coal Gas." Mr. W. J. Dibdin, "On the Estimation of the Illuminating Power of Gas Burners, especially those of large size."

On June 9th, Mr. W. S. Squire, "On the Processes Concerned in the Conversion of Starch into Alcohol, and their relation to Brewing and Distilling," illustrated.

CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE,

Monday, April 7th, 1884.

MR. DAVID HOWARD IN THE CHAIR.

The election of the local sectional committee took place, with the result that the committee as printed above were elected to serve for the next three years.

## THE ESTIMATION OF SULPHUROUS ACID IN ITS COMPOUNDS.

BY W. B. GILES AND A. SHEARER.

THE object of the investigation which forms the subject of the present paper was to examine the methods used in determining the sulphurous acid existing in the combinations of that acid which are of industrial importance. The method generally employed is that given by Fresenius in his "Quantitative Chemical Analysis," based upon Bunsen's well-known reaction between iodine and sulphurous acid in very dilute solutions. This method, if carried out strictly according to Fresenius's instructions, leaves little to be desired on the score of constancy of results, although they are generally a little low. The great objection to it, however, is the large dilution with air-free water which is necessary. It is almost impossible to obtain water *absolutely* free from air, and the difficulty of obtaining and preserving it as nearly pure as

possible is very considerable, and even where only an occasional analysis of sulphurous acid or its compounds is made involves much preliminary trouble and loss of time; while if a number of such determinations have to be made daily the difficulty becomes so great as to render a shorter and equally accurate process very desirable. The authors first investigated the means of estimating the sulphurous acid in the normal sulphites. Sulphite of sodium was selected as a type of these salts. The sample operated upon was prepared with great care from a crystallisation of several tons, and was the same for the entire series of experiments, it having been found hardly possible to prepare a sample sufficiently pure on the laboratory scale. The authors take this opportunity of thanking Messrs A. Boake and Company, of Stratford, for their kindness in affording them every facility in obtaining the samples, etc. The salt was first analysed gravimetrically, in order that a basis of comparison might be obtained, and, as will be seen from the following results, was nearly pure, having a composition closely agreeing with the formula  $\text{Na}_2\text{SO}_3 + 7\text{H}_2\text{O}$ . It may here be remarked that the authors' experience points to the foregoing formula as invariably representing the composition of crystallised sulphite of sodium. The salt mentioned by Muspratt, having the composition  $\text{Na}_2\text{SO}_3 + 10\text{H}_2\text{O}$ , has never been noticed by them during a lengthened experience of the crystallisation of the salt on a very large scale and under greatly varying conditions of temperature, concentration, and other influences such as acidity or alkalinity of the mother-liquors. These observations agree with those made by Rammelsberg and Schultz Sellack. The following are the details of the methods followed in the gravimetric analysis of the sulphite.

1.  $\text{SO}_2$  and  $\text{SO}_3$  were estimated by oxidising 0.5grm. of the sulphite with bromine water, subsequently adding HCl and heating, precipitating with  $\text{BaCl}_2$  after nearly neutralising the solution with  $\text{NH}_3$ , filtering, and igniting and weighing as  $\text{BaSO}_4$ .

0.5grm. of sulphite gave 0.465  $\text{BaSO}_4 = 31.93\% \text{SO}_3$ .

2. The  $\text{SO}_3$  existing as such was estimated in from 2grms. to 2.5grms. by the following process: A flask was filled with recently-distilled water, and fitted with a rubber stopper carrying a glass tube drawn out to a fine point. The water contained in the flask was then heated to boiling, and kept so for two hours, when 10c.c. of HCl free from  $\text{SO}_3$  were added, and the boiling continued for half an hour longer. The quantity of sulphite used was now weighed in a small test tube, and the stopper of the flask being momentarily withdrawn the tube and its contents were dropped in, the stopper then replaced loosely. When the evolution of sulphurous acid had somewhat subsided the cork was firmly fitted, and the boiling was continued till every trace of sulphurous acid was expelled. The excess of HCl was then almost neutralised by  $\text{NH}_3$ , and the  $\text{SO}_3$  was precipitated and weighed as  $\text{BaSO}_4$ . Two determinations made in this way gave the following results:—

- (a) 2.161grms. sulphite gave 0.025grm.  $\text{BaSO}_4 = 0.397\% \text{SO}_3$ .  
 (b) 2.594grms. sulphite gave 0.0305grm.  $\text{BaSO}_4 = 0.468\% \text{SO}_3$ .  
 The two determinations giving as a mean 0.400%  $\text{SO}_3$ .

3. By deducting the  $\text{SO}_3$  found in (2) from that found in (1), the  $\text{SO}_3$  resulting from the oxidation of  $\text{SO}_2$  is obtained, and from this the percentage of  $\text{SO}_2$  calculated.

	Per cent.
Total percentage of $\text{SO}_3$ (1).....	31.93
$\text{SO}_3$ existing as such (2).....	0.40
$\text{SO}_3$ resulting from oxidation of $\text{SO}_2$ .....	31.53
Equal to 25.22% of $\text{SO}_2$ or $\text{Na}_2\text{SO}_3, 7\text{H}_2\text{O}$ ....	99.32%

4. The water of crystallisation and moisture was estimated by drying 1grm. in the water oven. During



the desiccation, however, the salt was found to undergo a slight oxidation. The amount of oxygen so absorbed was found from the difference in the iodimetric titre before and after drying. The following results were thus obtained:—

1grm. dried at 100° lost.....	0.1985grm.
1grm. of the undried sulphite required.....	79.2 } c.c. $\frac{n}{10}$ I.
1grm. of the dried sulphite required.....	76.5 }
Difference .....	27c.c.

Calculated to oxygen the difference gives 0.0021grm. of oxygen absorbed during the drying, which amount has to be added to the observed loss of water, thus making the total loss of water 50.06 per cent.

5. The soda was calculated from the SO<sub>2</sub> and SO<sub>3</sub> found above, and the following figures embody the results obtained:—

Sample employed	Pure Na <sub>2</sub> SO <sub>3</sub> ·7H <sub>2</sub> O contains
SO <sub>2</sub> .....	25.22 .....
SO <sub>3</sub> .....	0.10 .....
Na <sub>2</sub> O .....	24.74 .....
H <sub>2</sub> O .....	50.06 .....
100.42	100.000

The sulphurous acid was next determined in another portion of the sulphite by the method given by Fresenius. Special precautions were taken to obtain air-free water for this estimation. The plan adopted was as follows: Two 60oz. flasks A and B were nearly filled with freshly distilled water, the contents heated to boiling, and the boiling continued for four hours. A rubber stopper, carrying a tube bent twice at right angles, one limb of the tube being short and the other long enough to reach to the bottom of the flasks, was now fitted to one of the flasks A, the short limb of the tube entering the flask and the long limb being plunged below the surface of the water in the flask B, the lamps being momentarily withdrawn. Matters so arranged, the lamp was placed below A and the boiling continued for an hour, the contents of the flask B being kept boiling by the steam passing through it. The boiling being then stopped the water in B was immediately covered with a layer of purified hydrocarbon oil to a depth of half an inch and the apparatus allowed to cool. As the cooling progressed a portion of the water in B receded into A, which was thus kept full and out of contact with air. A weighed portion of 0.5grm. of the sulphite was then dissolved in 250c.c. of the water from the flask A, and the solution poured with stirring into 40c.c. of  $\frac{n}{10}$  iodine solution, the whole operation being performed as quickly as possible to prevent absorption of oxygen from the air. It was found necessary to use 0.73c.c. of  $\frac{n}{10}$  thiosulphate to remove the excess of iodine. From this 0.5grm. of the sulphite consumes 39.27c.c. of  $\frac{n}{10}$  iodine, which calculated out gives 25.13 per cent. SO<sub>2</sub>, equal to 98.96 per cent. of Na<sub>2</sub>SO<sub>3</sub>+7H<sub>2</sub>O. The influence of dissolved oxygen in the water upon the results obtained, and also the extreme rapidity with which water takes up this oxygen, was well shown when a further determination was made with water from the same flask A, the stopper having been left out for a short time while the first trial was made, the same quantity of sulphite dissolved in the same quantity of water now giving 24.65 per cent. SO<sub>2</sub>, equal to 97.07 per cent. of Na<sub>2</sub>SO<sub>3</sub>·7H<sub>2</sub>O, or a loss of nearly 2 per cent. of sulphite in a few minutes. This very rapid change has been repeatedly noticed by the authors, and formerly sometimes led them to think that the variations between the iodine and the sulphite were caused by some irregularity in the reaction, until the true cause was detected. A num-

ber of estimations were then made, freshly distilled water being used instead of air-free water. In other respects the titration was carried out precisely as before.

TABLE 1.

Initials of Observers Name.	Sulphite used.	Water used.	C.c. of $\frac{n}{10}$ Iodine.	Na <sub>2</sub> SO <sub>3</sub> ·7H <sub>2</sub> O calculated per cent.
	Grammes.	C.c.	C.c.	
G.....	0.5	100.0	37.8	95.256
S.....	0.5	250.0	36.9	92.990
S.....	0.5	250.0	36.8	92.440
S.....	0.5	500.0	34.0	85.680
S.....	0.5	500.0	33.8	85.320
G.....	0.5	500.0	33.6	84.670
G.....	0.5	1000.0	27.6	69.550

As some observers, Mohr for instance, recommend the addition of bicarbonate, another trial was made, adding 2grms. pure potassium bicarbonate to 500c.c. of water, and dissolving *before* adding the sulphite.

This was found to require 33.60c.c. of  $\frac{n}{10}$  iodine, showing that the addition of bicarbonate has no effect in preventing the oxidation. After considerable thought and experience the following method was devised by the authors, in order to render unnecessary the use of air-free water, and was found to give highly satisfactory and concordant results. A portion of the finely-ground sulphite was introduced into an ordinary stoppered weighing tube, and the weight noted. A measured quantity of  $\frac{n}{10}$  iodine was then run into a beaker from a fine burette. In some of the experiments water in varying quantity was added for the purpose of establishing the influence this addition had upon the results. It was found, however, that though no precaution was taken to free the water from dissolved oxygen, the addition of quantities equal to the dilution found to be necessary by Bunsen in carrying out the titration of sulphurous acid upon iodine, viz., .05 per cent. SO<sub>2</sub>, had no influence upon the result, and also that the addition of water might be dispensed with altogether. A portion of the sulphite in the weighed tube was gently shaken out of the tube directly into the  $\frac{n}{10}$  iodine without previously dissolving it in water, stirring during the addition until the yellow colour just vanished; a few c.c. of starch then being added, and  $\frac{n}{10}$  iodine dropped in until the blue colour appeared (not more than a few tenths of a cubic centimetre were required). A second weighing gave the exact amount of sulphite used. The results are given in the following table:—

TABLE 2.

$\frac{n}{10}$ I run into beaker.	Water added.	Grammes of Sulphite used.	Total $\frac{n}{10}$ Iodine used.	Iodine calculated to SO <sub>2</sub> %	Iodine calculated to Na <sub>2</sub> SO <sub>3</sub> ·7H <sub>2</sub> O %
A { 50.0 c.c.	None.	0.63975	50.3 c.c.	25.16	99.07
{ 50.0 c.c.	None.	0.6385	50.1 c.c.	25.11	98.87
B { 49.9 c.c.	50 c.c.	0.6345	49.9 c.c.	25.17	99.10
{ 50.0 c.c.	50 c.c.	0.63825	50.2 c.c.	25.17	99.10
C { 50.5 c.c.	100 c.c.	0.649	51.0 c.c.	25.15	99.01
{ 49.8 c.c.	100 c.c.	0.636	50.05 c.c.	25.18	99.15
D { 50.6 c.c.	250 c.c.	0.6485	50.9 c.c.	25.12	98.89
{ 49.9 c.c.	250 c.c.	0.63225	49.9 c.c.	25.25	99.44
{ 49.5 c.c.	250 c.c.	0.6325	49.75 c.c.	25.17	99.10

The above figures show great accordance among themselves, and also with the percentages obtained both by the oxidation process and titration in diluted solution in air-free water. Throwing out the middle line in series D in the above table, the difference between the highest and lowest result is 0.07 per cent. of  $\text{SO}_2$ , or 0.28  $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ . The mean of the eight results (discarding the highest one of series D as being an error of observation) is 25.15 per cent. of  $\text{SO}_2$ , or 99.03 per cent. of  $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ . For comparison we tabulate the results by the three methods:

TABLE 3.

Method taken.	$\text{SO}_2$ % found.	$\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ found %.
By oxidation and subsequent precipitation with $\text{BaCl}_2$ , after deducting $\text{SO}_2$ found.....	25.22	99.32
By Fresenius's modification of Bunsen's method.....	25.13	98.96
By method above given the mean of eight determinations.....	25.15	99.03

Another modification of the above process, although based upon the same principle, gave slightly higher results, and is preferred by the authors, inasmuch as every chance of outside oxidation is removed from beginning to end of the operation by employing an excess of iodine to start with. In this case 0.5 grm. of the sulphite was weighed on a watch glass, and the glass and its contents transferred at once to a beaker containing 40 c.c. of  $\frac{n}{10}$  iodine, and the whole well stirred with a glass rod. In some cases the iodine was diluted with a measured quantity of distilled water, but, as in the former experiments, this dilution did not have any influence. The results thus obtained are shown in the following table:—

TABLE 4.

Grammes of sulphite used.	Cubic centimetres of water added to iodine.	Cubic centimetres of I taken.	Cubic centimetres of thiosulphate.	Cubic centimetres of I used (total).	$\text{SO}_2$ per cent.	$\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ per cent.	Initial of observer.
0.5	None	40.0	0.4	39.6	25.31	99.79	S.
0.5	None	40.0	0.4	39.6	25.34	99.79	G.
0.5	None	40.0	0.4	39.6	25.34	99.79	S.
0.511	None	40.4	0.0	40.4	—	99.61	G.
0.5	None	40.0	0.4	39.6	25.34	99.79	S.
0.5	50 c.c.	40.0	0.4	39.6	25.34	99.79	S.
0.5	200 c.c.	40.0	0.5	39.5	25.28	99.54	S.

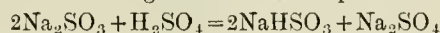
It should be mentioned that the observers worked with different balances, weights, burettes, and normal solutions, and in different laboratories. In the above experiments in no case was  $\text{HCl}$  used to liberate the  $\text{SO}_2$  before titrating, it being assumed that the  $\text{SO}_2$  would have a greater tendency to oxidise when in the free state than when combined. The following series of experiments in the presence of  $\text{HCl}$ , as recommended by Fresenius, hardly confirmed this opinion, but it will be seen that the results were very irregular, and that the use of this acid is not to be advised. (Table 5.) It should be mentioned that in the trials the sulphite was dissolved in the stated quantities of acid and water, and then immediately poured

into excess of iodine. The aeration of the water therefore accounts in some measure for the low results.

TABLE 5.

Grammes of Sulphite taken.	Cubic centimetres of $\text{HCl}$ 1160 used.	Cubic centimetres of water.	Cubic centimetres of $\frac{n}{10}$ I.	Cubic centimetres of thiosulphate.	Total cubic centimetres of Iodine.	$\text{SO}_2$ %	$\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ %
0.5	None	200	40.0	2.95	37.05	23.71	93.36
0.5	None	200	40.0	3.50	36.50	23.36	91.98
0.5	0.5 c.c.	250	40.0	1.80	38.20	24.41	96.26
0.5	0.5 c.c.	250	40.0	3.20	36.80	23.55	92.72
0.5	5.0 c.c.	200	40.0	2.20	37.80	24.19	95.25

The authors observed some years ago that when sulphite of sodium was titrated with normal acid, using cochineal as the indicator, the end point occurred when the following reaction was complete:—



the pink colour of the cochineal not being changed to yellow in the presence of  $\text{Na}_2\text{SO}_3$  until the latter is entirely converted into bisulphite—that is, while the normal sulphite reacts strongly alkaline to cochineal, the bisulphite is neutral, and free sulphurous acid is strongly acid to this indicator. The remarks of Mr. Thomson upon the action of acids upon the sulphites of sodium confirmed the authors' previous observations, while the indicator employed by him (methyl-orange) is decidedly more delicate. The following results were obtained by titrating the same sulphite as before used with  $\frac{n}{2}$  acid. In one set methyl-orange was used, in the other cochineal tincture.

TABLE 6.

Sulphite used, Grammes.	Cubic centimetres of water used.	Cubic centimetres of $\frac{n}{2}$ acid used.	$\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ Per cent.	Indicator used.
2.5	100.0	19.8	99.76	M. Orange
2.0	100.0	15.75	99.22	M. Orange
2.0	100.0	15.7	98.91	Cochineal
2.0	100.0	15.7	98.91	Cochineal
2.0	200.0	15.7	98.91	M. Orange
2.0	200.0	15.65	98.59	Cochineal
2.0	500.0	15.40	97.02	M. Orange
1.0	250.0	7.7	97.02	Cochineal

The influence of the dissolved oxygen is thus as clearly established in the above trials as when iodine was used under like conditions of dilution. In both cases the percentage of sulphite rapidly diminishes with the increased volume of water employed.

## SUMMARY.

The method of procedure recommended by the authors for the general estimation of sulphurous acid in its compounds may be briefly summed up as follows: The sulphite, if solid, is weighed out on a watch glass,



and introduced at once *without dissolving it in water* into a known *excess* of decinormal iodine; after stirring till the reaction is complete, which takes some little time when insoluble sulphites (*e.g.*, calcium sulphite) are dealt with, the excess of iodine is determined in the usual way by means of decinormal thiosulphate and starch; a second experiment is then

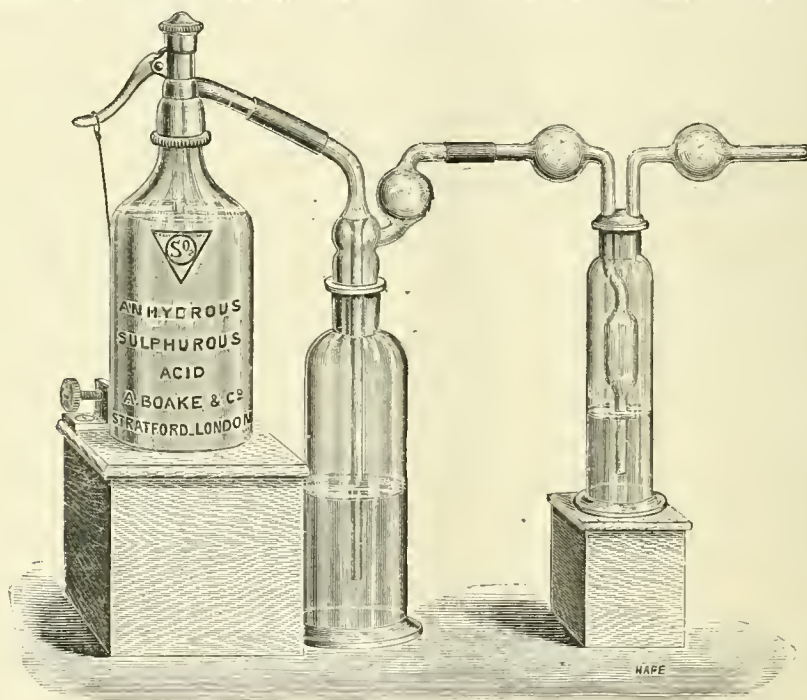
made, in which a *slight* excess only of the  $\frac{n}{10}$  I, as indicated by the first trial, is employed, and the titration carried out as before. The writers in the foregoing remarks have only instanced the sodium salt, but they have found the method equally accurate for the estimation of concentrated sulphurous acid solution and bisulphites, the reverse action of  $\text{SO}_3$  upon hydriodic acid noticed by Bunsen not affecting the result. The following compounds of  $\text{SO}_2$  have been repeatedly analysed in the foregoing way, and have given highly concordant results with those obtained by the much more tedious gravimetric method: Solution of  $\text{SO}_2$ , specific gravity 1.067 at  $11^\circ \text{C}$ ., containing 13.1 per cent. of  $\text{SO}_2$ ; bisulphites of sodium (solid),

hours or more. By simply tightening the small screw more or less, any desired flow of gas can be obtained and regulated, and one of these siphons will evolve about 500 litres of  $\text{SO}_2$  gas.

#### DISCUSSION.

Dr. ARMSTRONG said it was extremely desirable to have not only simple but accurate methods of analysis. This method of Messrs. Giles and Shearer had both these merits, as he could say from comparative tests in his laboratory, in which the results were concordant and accurate. With regard to the siphon of sulphurous anhydride he found it extremely handy and of especial value where many iron determinations had to be made.

Mr. B. E. R. NEWLANDS regarded the siphon arrangement for sulphurous acid gas as a valuable adjunct in the laboratory, having employed it for some time. With regard to the authors' method, he was of opinion it was more especially useful where numerous estimations had to be rapidly made. For ordinary and occasional work the old method of oxi-



potassium, ammonium, calcium, magnesium, zinc, etc., some of these bodies containing over 60 per cent. of  $\text{SO}_2$ ; the sulphites of sodium, ammonium, calcium, magnesium, aluminium, zinc, etc. In the case of fluids, the solutions are sealed up in small, thin blown glass bulbs, and broken under the iodine when loss of gas is apprehended. The solutions are previously cooled in a freezing mixture. The writers, in conclusion, throw out the suggestion that the very rapid absorption of oxygen in water by normal sulphite of sodium may be worthy of attention as a means of determining dissolved oxygen in water analysis. They have also pleasure in bringing to the notice of the Section the very convenient form in which anhydrous sulphurous acid liquefied is now offered by Messrs. A. Boake and Company. The small apparatus shown at work has been of great service to them in their investigations, as it is capable of affording a steady stream of pure  $\text{SO}_2$  gas for forty

hours or more. By simply tightening the small screw more or less, any desired flow of gas can be obtained and regulated, and one of these siphons will evolve about 500 litres of  $\text{SO}_2$  gas.

hours or more. By simply tightening the small screw more or less, any desired flow of gas can be obtained and regulated, and one of these siphons will evolve about 500 litres of  $\text{SO}_2$  gas.

#### FURTHER NOTES UPON THE SOLIDIFICATION OF LIQUID FATTY OILS.

BY WM. LANT CARPENTER, B.A., B.SC., F.C.S.

At the meeting of this Section, in March, 1883, I gave a somewhat detailed account, which appeared in the

Journal of the Society for March 29th of that year, of the ingenious process of M. St. Cyr Radisson, of Marseilles, for converting liquid oleic acid (the chief residue in the manufacture of stearic and palmitic acid from neutral fats, such as tallow, palm oil, etc.) into solid crystalline palmitic acid. The process is founded on the laboratory reaction announced in 1841 by Varentrapp, that when oleic acid was heated with a great excess of caustic potash it was resolved into palmitic acid, acetic acid, and hydrogen, or

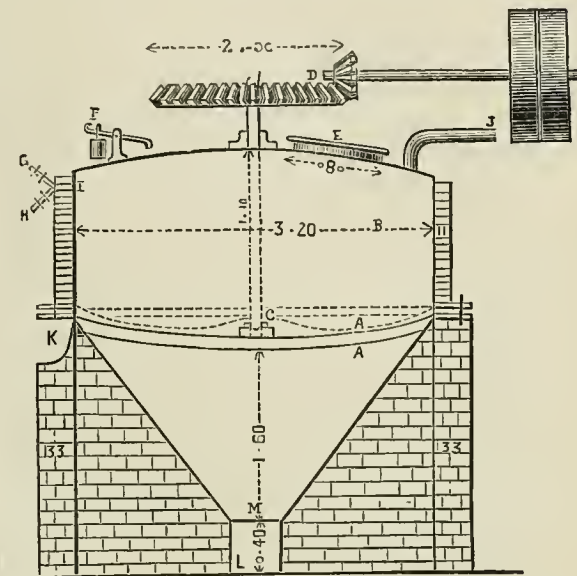


Regarded from the point of view of this Society the process may be shortly described as that of a fusion with excess of caustic alkali at a high temperature, and it is in the practical carrying out on a large scale of this difficult laboratory operation that the special interest of the process consists.

In the discussion which followed my paper I was asked some questions of detail as regards the apparatus employed, which I was then unable to answer; but having in August last spent several days in Marseilles with the inventor, I propose now to give a little further information on the subject. The process was patented in most European countries, and in the United States, the English patent being June 9th, 1869, No. 1782, and the American, April 12th, 1870, No. 101935; but a variety of causes have prevented M. Radisson from making known his invention as widely as it deserves. The vessel in which the fusion takes place is technically called a "cartouche," and is shown in section in the annexed figure. It is a cylindrical vessel A B, 3.20 metres in diameter and 1.30 metre in height, the two ends being slightly dished, and the bottom made of cast iron, while the sides are of wrought iron. The whole is set in brickwork in such a way that the bottom is at least 1.75 metre above the fire-bars M, thus giving a large fire-chamber, a very important point in the facility for control of temperature and maintenance of a uniform heat. The cartouche contains any convenient mechanical agitator C, worked with gearing D, also a manhole E, safety valve at F, steam and water cocks G, H, exit pipe J, for steam and hydrogen, and a large outlet pipe K, with suitable valve for removing the fused mass at the end of the operation. In working with caustic potash, the original form of the process, the cartouche is charged with 1,500 kilos. of oleic acid and 2,500 kilos. of caustic potash ley of specific gravity 1.400. Heat is applied, and when the disengagement of steam ceases, the manhole E is closed, and the evolved gases pass through J to a coke-tower condenser, and thence to a gasometer or gasholder. At 290° C. decomposition commences and much hydrogen is evolved. From 300° to 310° C. is the proper temperature for the operation, and at 320° the odour of the evolved gas changes, and the operation is then suddenly arrested by injection of steam into the mass, by the Giffard injector I, in order to avoid destructive distillation. From 36 to 40 hours are required for one complete operation in the cartouche, including filling and emptying. The progress of the operation is judged by taking samples, which are decomposed, and tested for the melting point of their fatty acids. The method employed is that of Dalican, universally adopted in France, which gives really the "point de figeaison," or congealing point of the fatty acid, within 0.1° or 0.2° C.

Writing in 1878 a short account of his enormous candle factory and of the objects exhibited for the Industrial and International Exhibition in Paris, M. Fournier says of this process, "La maison exploite depuis trois ans le système Radisson dans une annexe de son usine, et chaque jour elle transforme 2 à 3000 kilogrammes d'acide oléique en acide palmitique. La stéarinerie a donc pris possession d'une manière

définitive de ce nouveau procédé si important pour son avenir. Cette transformation est la nouveauté



A. Cast-iron bottom, 3.20 metres diameter. B. Wrought-iron cylinder. C. Agitator. D. Gearing. E. Manhole, serving also as a charging hole. F. Safety valve. G. Steam cock. H. Water cock. I. Giffard's injector. J. Pipe to coke tower. K. Outlet for palmitate of potash. L. Ashpit. M. Fireplace.

la plus saillante qui se soit produite dans l'industrie stéarique depuis l'Exposition de 1867."

### CORRECTION.

BRISTOL WATER.—In his paper "On the Filtration of Potable Water," which was published in the March number of the Journal, Mr. S. H. Johnson includes Bristol among the towns which "are still supplied with sewage-contaminated water from rivers and surface wells." We are informed that the water supplied by the Bristol Waterworks Company is not supplied from sewage-contaminated sources, but "from copious springs on the north side of the Mendips, and from a well at Chelvey, from which places the water is conveyed in culverts and pipes the whole distance, and is therefore entirely free from surface contamination."

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Notices of papers and communications for the meetings to be made to Edward George Ballard, Queen's Park, St. Helens.

The next meeting of this Section will be held on Wednesday evening, 7th May, 1884.



UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL,

April 2nd, 1884.

PROF. J. CAMPBELL BROWN IN THE CHAIR.

## INJURY DONE TO VEGETATION BY GASES ARISING FROM CERTAIN MANUFACTURING PROCESSES.

BY SIEGFRIED HAMBURGER, PH.D.

THE question of damage done to vegetation by smoke and acid gases escaping from the chimneys of manufacturing establishments is comparatively of recent date, and complaints seem to have increased with the progress which industry has made in the last decades. Such complaints have principally been brought forward in districts where many manufactures were carried on, and the blame was always laid at the doors of chemical manufacturers, and especially the alkali makers, of whose works it was known that they gave rise to large quantities of acid gases. Lately this subject has been taken up by several unprejudiced investigators, and great credit is due to them for having shown that gases arising from other manufactures may be equally noxious, and even more so. Much, however, still remains to be done in that direction. People somehow cling to their old belief, and every endeavour ought to be made to dispel that erroneous notion. That is the object of this paper. I shall give you the results of analyses and experiments I have made, and the conclusions I have drawn from them. But I wish to remark here that the samples of leaves, grasses, etc., used for my purpose were collected in a somewhat casual way while I was taking a walk, and without a view to the publication of the results of my investigation. Thanks are also due to several friends of mine who have sent me specimens of leaves from different parts of this country.

Some time ago Mr. R. Hasenclever, of Aachen, the well-known director of the Rhenania Alkali Company, published in the German *Chemische Industrie*\*, an excellent paper on the same subject, and I shall give you some of his statements. Dr. Angus Smith, who is perhaps the greatest living authority on chemical climatology, treats this matter in his well-known book on "Air and Rain," and I have taken some quotations from that work.

At the outset permit me to state in what way my analyses and tests were carried out. On seeing in a field, or on trees, grasses or leaves whose appearance seemed to indicate that they had been attacked by noxious vapours, I tried their surface for the reaction upon damp litmus paper, and tested them afterwards in the laboratory for  $\text{SO}_3$  and Cl. Sometimes I analysed plants which were looking healthy, but were growing in the immediate neighbourhood of suspicious specimens, so as to compare the results. Only four times out of several hundreds of tests with litmus paper did I find a distinct acid reaction. We shall see later on that tests with litmus paper are misleading. The analysis was conducted by saturating the weighed substance with a weak solution of pure  $\text{Na}_2\text{CO}_3$ , boiling down to dryness, and incinerating the residue in a platinum crucible. The ash was lixiviated with water, and the filtered solution divided into two equal parts, in one of which  $\text{SO}_3$  was determined,

and in the other Cl was estimated. On other occasions I lixiviated the substance with a weak solution of  $\text{Na}_2\text{CO}_3$ , filtered and evaporated to a smaller bulk. After dividing the extract into two equal parts, I tested one for  $\text{SO}_3$  and the other for Cl in the usual way. The leaves were then dried and incinerated in a crucible, adding some  $\text{CO}_3\text{Na}_2$ . The ash was treated as mentioned above.  $\text{SO}_3$  and Cl are given in the table below, as well as ashes, all calculated for 100 parts of the dry substance of the plants. I should just like to mention that the St. Helens specimens, from 1 to 13, were taken from a part of the neighbourhood which is most frequently exposed to the smoke of alkali works.

Now you will at once observe that even in the same localities the amount of  $\text{SO}_3$  and Cl is very variable. Furthermore, while the leaves of one plant, with a considerable amount of both constituents, gave an acid reaction, those of another, with the same amount and sometimes even more, gave a neutral reaction, showing clearly that *the presence of free acid is no proof that the organism of the plant is saturated with acid*. It seems very strange, and it does not allow of easy explanation, that a plant with so large quantities of  $\text{SO}_3$  and HCl can show a neutral reaction, provided that these acids were originally deposited in an uncombined state. However, this must not be the case. Indeed, we have much reason to suppose that the Cl was deposited on the plant combined with Na as NaCl, but the same cannot be said of the  $\text{SO}_3$ . As there are only traces of ammonia present in the extract of leaves, it is difficult to see what strong basic compounds have acted upon  $\text{SO}_3$  so as to rob it of its acid nature. In "alkali towns" we may perhaps assume that alkaline matters are present in the air, which have been mechanically carried away from the black ash chimneys. But this explanation will not hold for such towns as Derby, for instance (*vide Anal.* 20 and 21). Objections might be raised that my analyses do not represent a fair average of specimens, as they are taken in fine weather. But I really do not think it matters much, as any free acid, accumulated by condensation, etc., etc., would be washed down into the earth by subsequent rain, and thus escape detection. Besides, the analyses go to show that a small amount of free acid does no harm to the plant which absorbs it in a way still unknown to us. The same will, perhaps, also explain why I did not find as much Cl as we should have expected. I leave the question open, and would be glad to hear the opinion of members on this point.

Mr. Hasenclever, in giving the results of some twenty analyses of leaves taken from trees from different localities, unfortunately does not state the reaction of the leaves which he submitted to analysis, nor the appearance and health of the trees from which the samples were taken. This omission is all the more regrettable, as the outward appearance, *when properly interpreted*, gives most important indications in our present imperfect state of knowledge of this subject.\*

\* Since writing this paper Mr. Hasenclever has been kind enough to inform me of the reason why he did not state the reaction of the leaves or the watery extract respectively. He says: "According to my experience, and to the investigations of Prof. Freytag, of Bonn, and Prof. von Schroeder, of Tharand, we are not entitled to draw the conclusion that a plant must be saturated with free acid because it has an acid reaction on its surface." I certainly agree with him in that, and I have already taken an opportunity of pointing this out in the remarks following the results of my analyses. But I thought it important to give the reaction of leaves, just in order to show that in leaves with an acid reaction the quantity of  $\text{SO}_3$  and Cl may be smaller than in those with a neutral reaction. Like other investigators, I did not think it necessary to make a separate determination of the free acid. Mr. H.'s arguments on this point well deserve attention. He proceeds to say that in a lawsuit for damage done by the smoke, the following technical evidence was given: "The estimation of

\* *Chem. Industrie*, 1879, p. 225 and 275.

To return to my analyses. You will also see that on the whole the amount of  $\text{SO}_2$  exceeds that of Cl, in some cases even considerably. It would be wrong to assume that all Cl found in the plants was originally present as HCl. It is well known that our ordinary fuel, coal, contains Cl as NaCl, but there is no Cl to be found in coal-ash, and it is only reasonable to infer that the NaCl has been driven out with the other volatile constituents. It has also been proved that in non-manufacturing districts, NaCl has been found in the rain, undoubtedly produced by coal burnt in household fires. In industrial districts this will be all the more the case. In some instances, as previously indicated, the presence of NaCl may be accounted for by the fact that some alkali makers allow the escape of their HCl condensers to go into the black-ash chimneys, which mostly have an alkaline reaction.

We will now consider the  $\text{SO}_2$ . The amount of this gas escaping from alkali works or their O.V. plant respectively is so small as certainly not to be in proportion to that found in different plants. The  $\text{SO}_2$  must therefore be generated in another way, and in the case of St. Helens I have only to allude to such industries as glass works and copper works. But a far larger amount is produced by the consumption of coal as fuel. I may safely say that the coal used in works for firing contains on an average 2 per cent. of volatile sulphur—as a rule as pyrites, “coal brasses”—which on burning give off  $\text{SO}_2$ . To give you an idea of the amount usually present in the air, I quote the figures of Dr. Angus Smith, who calculates that in a part of London, where coal is only consumed for domestic purposes, a million cubic feet of air contains 730 grains of  $\text{SO}_2$ . At Manchester, where coal is largely consumed for industrial purposes, the figure rises to 1,098 grains of  $\text{SO}_2$ . I found at St. Helens, as an average of several determinations, 1,260 grains of  $\text{SO}_2$  in one million cubic feet of air. Mr. Fletcher calculates (in 1879) as gases escaping into the air of St. Helens *per week* :—

From fire gases.....	800 tons $\text{SO}_2$
From copper works.....	380 tons $\text{SO}_2$
From glass works.....	180 tons $\text{SO}_2$
From alkali works.....	25 tons HCl

That is to say, the alkali makers turn out less than one-seventh of the amount of the glass works and less than one-fifteenth of that of the copper works. I have calculated\* to what figure the percentage of fire gases for  $\text{SO}_2$  may rise, if we burn coal of the following average composition :—

	Per cent.
Carbon .....	67.4
Hydrogen .....	4.1
Oxygen .....	6.6
Nitrogen .....	1.2
Water .....	2.0
Volatile Sulphur .....	2.0
Ash, etc. ....	16.7

Suppose we burn such a coal with just the necessary

free acid in the extract of leaves does not warrant the conclusion that the leaves contained free acid. Aqueous extracts of plants quickly undergo decomposition, giving rise to the formation of organic acids. The extract of beech and oak leaves examined for free acid showed, that the extract of beech leaves from the Kettenforst (a neighbourhood free from smoke of chemical works), contained as much free acid as that of the leaves of the same kind of tree from the “Kleine Probststei” (in the neighbourhood of chemical works). The same was the case with oak leaves.” Prof. von Schroeder and Carl Reuss say in their exhaustive book on “Damage Done to Vegetation by Smoke”: “The extraction of leaves with water is a clumsy process: it can never be uniform or complete. The  $\text{CO}_2$  interferes with the method, and the extract undergoes decomposition even at ordinary temperatures, and at the boiling point, quickly enough to allow alterations to go on in the percentage of free organic acids. To prove injury done by smoke the determination of free acid by itself is worthless in every case. Still more faulty are those statements, often used in evidence, that the injured plants gave a very strong acid reaction with litmus paper.”

\* *Industrie-Gase* v. Dr. Clemens Winkler.

quantity of air, avoiding any excess whatever, then 100lb. of coal will change as follows :—

67.4lb. C require 179.7lb. O and yield 247.1lb. $\text{CO}_2$ .	
4.1lb. H require 32.3lb. O and yield 36.9lb. Steam.	
1.2lb. N	yield 1.2lb. N.
2.0lb. $\text{H}_2\text{O}$	yield 2.0lb. Steam.
2.0lb. S	yield 4.0lb. $\text{SO}_2$ .

100.0lb. of coal require 214.5lb. O and yield 291.2lb. fire-gases.

We have now to deduct 6.6lb. O found in the coal from 214.5lb. O required, and we have to take from the air  $214.5 - 6.6 = 207.9$ lb. O, where we find it mixed with

$$\frac{207.9 \times 76.8}{23.2} = 688.2 \text{ lb. N}$$

which increases the amount of fire gases to  $291.2 + 688.2 = 979.4$ lb., consisting of—

	Vol. in cub. ft. 0° C. and 760mm. B.*
247.1lb. $\text{CO}_2$ .....	2008.4
36.9lb. Steam .....	733.4
1.2lb. N .....	15.3
2.0lb. Steam .....	39.7
4.0lb. $\text{SO}_2$ .....	22.4
688.2lb. N .....	8791.6
	11610.8

The fire gases would therefore contain—

$$\frac{22.4 \times 100}{11610.8} = 1.93 \text{ vol. per cent. of } \text{SO}_2$$

However, in burning on the large scale, we have to dilute the gases with an excess of air, in order to bring about complete combustion. Further, when they reach the atmosphere at the top of the chimney, they at once mingle with air, by which a considerable dilution takes place. Notwithstanding all that, we must not forget that the classical investigations of Professor Freytag, have proved, that air containing more than .003 volume per cent. of  $\text{SO}_2$  will do injury to vegetation. That would mean to bring down undiluted fire gases to such a low percentage of  $\text{SO}_2$ , they must be mixed with 64 times their own volume of air, and I do not believe that we can reach that degree of dilution in crowded manufacturing districts, *when the atmosphere is in a moist state.* Besides, cold and rain, etc., produce condensation—that is, concentration of the acids, and at such times the vegetation must be affected. We can therefore conclude that it is not HCl which does the damage, but  $\text{SO}_2$  produced by coal burning, etc., and the analyses 20 + 21 in my table, compared with No. 22, support this point of view. I shall give you another striking illustration of my assertion just made in two examples, mentioned by Professor Stoekhardt, in the *Tharander Forstliches Jahrbuch*.† In a farmyard, there were near the chimney belonging to a boiler fire two chestnut trees, which had been able to withstand for several decades the smoke of oak-wood serving for fuel. After changing the fuel from wood to coal, the trees commenced at once to show signs of decay and died in the second year, the young leaves getting killed soon after they appeared. At another place an orchard was completely destroyed, when a brick furnace which had been supplied with wood for 20 years was rebuilt and arranged for coal firing. I could add a good many more examples, which would only serve to prove the same, namely, that the carrying on of large industries, or more accurately the consumption of much coal in a small district, is incompatible with a flourishing vegetation. Mr. Hasenclever and certain public bodies who have carefully inquired into the question of injury to vegetation have come to this conclusion. They admit that damage is done—in fact, nobody can deny it—but

\* It seems rather strange to speak of steam at 0° C., but it makes no difference in the result whether we assume the gases at 0° C. or 150° C., or any other temperature.

† *Thar. Forstl. Jahrbuch*, Vol. 21, page 31.



No.	Specimen taken at	Kind of Plant.	Reaction of Leaves.	Per 100 Dry Substance.			Remarks on Appearance, etc.
				SO <sub>3</sub>	Cl	Ash.	
1	St. Helens { after some days } opposite { ( of damp weather } two large alkali works {	oats	slightly acid	2.89	.80	11.31	bad crop; yellow corrosions on edges.
2	St. Helens { after some days } large ( of fine weather } alkali works {	grass	neutral	2.21	.69	10.37	discoloured.
3	St. Helens { about 1 mile off alkali } ( works, Parr {	clover	neutral	2.56	1.13	9.38	fair crop; leaves good.
4	St. Helens { about 1 mile off alkali } ( works, Parr {	grass	strongly acid	2.09	1.21	11.29	corroded over the whole surface.
5	St. Helens { about 1 mile off alkali } ( works, Parr {	potatoes	neutral	2.53	1.16	9.06	good crop; leaves had no marks.
6	St. Helens { about 1 mile off alkali } ( works, Parr {	apple tree	slightly acid	1.49	.65	9.40	yellowish-red corrosions; tree nearly destroyed.
7	St. Helens { about 1 mile off alkali } ( works, Parr {	apple tree	neutral	1.86	.61	10.22	good; few marks on leaves; corrosions.
8	St. Helens { about 1 mile off alkali } ( works, Parr {	potatoes	strongly acid	3.26	.82	9.09	good crop; leaves brown corrosions?
9	St. Helens { about 1 mile off alkali } ( works, Parr {	potatoes	neutral	2.89	1.12	9.35	good crop; leaves had no marks.
10	St. Helens { about 2 miles off alkali } ( works, Garswood, be- tween Wigan and St. Helens {	turnips	neutral	2.56	1.01	10.30	leaves corroded.
11	St. Helens { about 2 miles off alkali } ( works, Garswood, be- tween Wigan and St. Helens {	hedges	neutral	1.09	4.02	11.59	yellow-brown corrosions; decaying.
12	St. Helens { about 2 miles off alkali } ( works, Garswood, be- tween Wigan and St. Helens {	beech	neutral	2.19	1.33	10.34	yellow-red corrosions.
13	St. Helens { about 2 miles off alkali } ( works, Garswood, be- tween Wigan and St. Helens {	beech	neutral	1.36	2.04	11.36	tree higher than the surrounding ones; top destroyed, probably owing to exposure to wind and weather; leaves had no marks.
14	St. Helens—St. Ann's	rhododendron	neutral	1.60	1.59	7.01	leaves discoloured and much corroded, nevertheless the plant was flowering very well.
15	St. Helens—Windlehurst	holly	neutral	2.01	.30	8.61	good.
16	St. Helens—Crank	elm	neutral	2.40	1.06	11.21	yellow-brown corrosions, tree decaying. It was standing by itself, without any shelter whatever.
17	St. Helens—Moss Bank	hedges	neutral	2.86	1.17	11.12	} good.
18	Prescot { Knowsley Park {	oak	neutral	.81	.19	8.38	
19	Prescot { Knowsley Park {	elm	neutral	1.16	.31	11.11	} good.
20	Derby { part of the town exposed } ( to smoke of works {	poplar	neutral	1.69	.29	12.71	
21	Derby { part of the town exposed } ( to smoke of works {	apple tree	neutral	1.63	.39	8.22	leaves much corroded; tree decaying; the top already de- stroyed.
22	Derby—no works	poplar	neutral	.76	.09	14.08	tree decaying.
23	Neighbourhood of London—no works	shrubs	neutral	.98	.24	13.61	} good.
24	Liverpool—Sefton Park	holly	neutral	1.12	.20	7.38	

the question of remedy is a most difficult one, and it is not easy to approach it without interfering with the natural development of industry. For this reason I cannot agree with those who suggest opposition to the accumulation of works in one place or district, as experience goes to show that smoke does no harm to vegetation if in proportionally small quantities. The technical means which have been devised to prevent or at least to lessen the damage have not been successful up to the present. It is not sufficient to insist upon chimneys being built high in crowded industrial districts. They do not dilute the gases enough in damp weather, especially if the quantity is very large, as is always the case in manufacturing towns. They may do very well for isolated establishments. The washing of smoke is still in its infancy, and we have to overcome a great many mechanical difficulties before we can consider that matter seriously. If we ever solve the problem of condensing diluted acid gases, then we may confidently hope to remove the greatest enemy of vegetation— $\text{SO}_2$ . That manufacturers are not opposed to condensation, as is often asserted, is best proved by the example which the alkali-makers have given to the public. They certainly deserve to be complimented on the manner in which they have brought to perfection the condensation of  $\text{HCl}$ . In spite of many most expensive failures, they continued their experiments till they succeeded, and it is therefore neither fair nor reasonable to harass them with unfounded accusations.

Besides acid gases, there are a good many other causes of the decay of trees, such as exposure to wind and weather, drainage of the forest, or the removal of fallen leaves. But as these do not concern our subject, I am satisfied with only mentioning them. However, one ought to ascertain whether those reasons were not the immediate cause before hastily forming the conclusion that the damage had been done by noxious gases.

All plants do not suffer in the same degree from acid gas; fruit-trees are most sensitive to it, while forest trees have a stronger constitution. Agricultural plants show a still more powerful resistance. Those who take an interest in this matter will find a table, indicating the 'scale of injury,' at the end of Dr. Angus Smith's book on "Air and Rain." Some people contend that the soot in the smoke may damage vegetation by choking up the pores of plants, but Prof. Stoeckhardt has proved by direct experiments that this is not the case, even if the quantity of soot is so great as to give the tree a coal-black appearance. All other conditions being favourable, the tree continues growing, as if nothing had taken place.

I shall now give you the result of some experiments which I made, in order to find out what amount of  $\text{SO}_3$  and  $\text{HCl}$  a certain kind of a plant can take up without suffering from corrosions, and what reaction within the plant brings about its decay. I exposed

leaves to the action of  $\frac{n}{10}$ ,  $\frac{n}{100}$ ,  $\frac{n}{1000}$ , and  $\frac{n}{2000}$   $\text{SO}_4\text{H}_2$  and  $\text{HCl}$  of equivalent strength. The normal acids produced discolouration in less than half an hour, yellow-brown spots appearing in the middle of the leaves, and extending gradually over the whole surface. The  $\frac{n}{10}$  was also too strong, although it took about three hours before the first signs of destruction appeared; the  $\frac{n}{100}$  acid acted in about a day, but already here the action of  $\text{SO}_4\text{H}_2$  was stronger than that of  $\text{HCl}$ . The  $\frac{n}{1000}$   $\text{SO}_4\text{H}_2$  produced discolouration in about a week. The  $\text{HCl}$  of

equivalent strength required 10 days. An  $\frac{n}{2000}$  acid ('0245 grm.  $\text{SO}_4\text{H}_2$  and '01825 grm.  $\text{HCl}$  per litre) seems to have no action. Two green-house plants were submitted to a daily spray of the  $\frac{n}{2000}$  acids, and showed no corrosion after one month. With different specimens time and concentration of the acids will probably have other consequences. But having not sufficient time at my disposal I had to discontinue these observations.

With regard to the reaction which is going on within the plant, I prepared an alcoholic solution of chlorophyll by digesting green leaves with absolute alcohol. The solution retains its green colour for a long time when kept in a stoppered bottle, but changes to a dirty-brown, and gradually deposits a brown precipitate, when an acid acts upon it. This takes place at once with an acid of moderate strength, but with a very dilute acid it requires some time. On the surface of corroded leaves we meet with a similar discolouration, and if we adopt the theory that it is the chlorophyll which decomposes the carbonic acid, we can well understand that a plant must die if the destruction of the chlorophyll has extended over a considerable part of the leaves. The corrosion of leaves brought about by acid gases can be compared with an inflammation of the lungs in the human body.

Let us now look at the value we may give the results of chemical analysis. Dr. Angus Smith holds "that in many cases we might be able to show by analysis that acid gas has been the cause of injury," but I can find no analyses in his publications which support his opinion. I cannot quite agree with him, because I found the same amount of  $\text{SO}_3$  and  $\text{Cl}$ , and even more in healthy plants than in decaying ones (*vide Anal.* 6 and 7). Mr. Koenig\* has had the same experience, and found more  $\text{SO}_3$  in a healthy-looking larch tree than in a diseased one. I think a different state of the atmosphere exercises a different action, other things remaining the same. We may perhaps be able to prove conclusively injury caused by a certain gas from a particular works, if we happen to catch the right moment, which is exceedingly difficult. The utmost precaution is necessary before pronouncing a decisive opinion. Nevertheless, if we continue to study this matter, we shall obtain valuable information, and for this reason I wish to give some indications to chemists who may be called upon to investigate in such cases. One commences by collecting in the immediate neighbourhood of the district in which complaints arise suspicious specimens as well as healthy ones, which are then to be compared chemically with each other. The outward appearance alone is no criterion for injury done by gas, as I often saw marks on the surface of leaves which had been caused by fungi and insects in an undeveloped state, and which were just like marks from acid corrosion. It would be advisable to examine all specimens by the microscope. Plants from other parts of the country, under the same climatic conditions, may also serve for comparison. Besides, the "scale of injury" is a very important indicator; and if, for instance, fruit-trees are less damaged than forest-trees or corn, it is very improbable that the injury was done by smoke. However, one must notice whether the fruit-trees had not some sort of shelter which kept off the gases, as practical farmers have often observed, especially with corn, that seemingly unimportant causes, such as a small elevation, hedges, etc., were able to protect their property from the influence of noxious gases. Further, we would have to test the air and



rain for acidity, making allowance for climatical changes; and better still, wherever possible, to make direct experiments with gases under the same conditions as complained of, and observe the reactions. The chemical investigation will chiefly consist of estimating  $\text{SO}_3$  (I never found  $\text{SO}_2$  in the extract) and Cl. Other requirements for special cases will suggest themselves to the investigator, and I should be pleased, if other gentlemen would take up this matter and publish their results for the benefit of the much harassed chemical manufacturers. The literature on this subject is very poor indeed, and more information is badly wanted. I shall myself continue my observations, and may have an opportunity of giving you further results at some future date.

I conclude with the words of Mr. Hasenclever: "The blame for injury done to vegetation can be brought home more or less to all manufacturing establishments; but one must not forget that this is only a small nuisance compared with the great economic advantages mankind owes to the enterprising spirit of industry. A great improvement has already taken place, and Dr. Angus Smith, in speaking of the former and the present state of things, considers them as different as barbarism and civilisation."

Owing to the late hour the discussion was postponed until the next meeting.

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MANCHESTER TECHNICAL SCHOOL,

April 1st, 1884.

MR. IVAN LEVINSTEIN IN THE CHAIR.

## ON THE OXIDATION OF CELLULOSE, WITH SPECIAL REFERENCE TO THE CHEMISTRY OF BLEACHING AND PRINTING.

BY C. F. CROSS AND E. J. BEVAN.

THE chemical activity of cellulose, in those forms best known to the technologist, is not of a pronounced character—indeed, it may be defined as an inert substance. Were it not for this absence of affinity, which is a distinguishing characteristic of cellulose, it could not play the important part which it does in the chemical laboratory. As it is, however, cellulose is the almost universal medium of separation by way of filtration, and in point of usefulness stands, perhaps, only second to water. This important function of

cellulose is obviously conditioned by its inability to enter into stable combination with the acids, bases, and salts with which inorganic analysis has to do. On the other hand, it is a matter of experience that there does exist in cellulose a certain power of attraction for these compounds, and this attraction is considered by certain investigators to be of a truly chemical nature, *i.e.*, to result from the structure of the cellulose molecule itself. Thus Mills and Takamine have shown (*Chem. Soc. Journ.*, 1883, 153) that cotton, when immersed in dilute solutions of hydric chloride and of sodium hydrate, concentrates these compounds within itself to an appreciable extent, the ratio in which they are respectively attracted being of a simple molecular character, *viz.*,  $3\text{HCl} : 10\text{NaOH}$ . It is also worthy of notice that the ratio determined for silk was the same, whereas for wool it is  $2\text{HCl} : 3\text{NaOH}$ . This attraction, however, is of so feeble a character that it is overcome by water, and when it is considered that the ultimate visible structure of these fibres must play some part in the result, it is difficult to say to what extent it is not due to mere osmotic differentiation. On the other hand, it is not for us to imagine, still less to assign, a mutual limit to the action of the so-called chemical and physical forces. Rather we would join in the anticipation that the investigation of bodies like cellulose may contribute to the breaking down of the barrier erected by classification, in showing that osmose is a truly molecular phenomenon.

Passing from the laboratory to the factory, experience of the numerous applications of cellulose, notably in the textile arts, teaches us that it has an activity which can be evoked under suitable treatment, and it is to certain operations of the bleacher and colourist that we wish to direct attention from a point of view that has hitherto been conspicuously neglected. Some time since, with the view of throwing light upon the constitution of cellulose, we made a study of its oxidation by nitric acid. Purified cotton was boiled with the strong acid (50 per cent.) for several hours, and the solution, containing a large quantity of oxalic acid, was filtered from the insoluble residue. This amounted to about 30 per cent. of the weight of the original cotton, and took the form of a fine white powder, which, however, on washing, was converted into a gelatinous mass. In this form it was entirely soluble in dilute alkalis, and was precipitated from such solutions in a form resembling pectic acid on the addition of acids, alcohol, or saline solutions. This substance, which we provisionally termed oxycellulose, was prepared by similar treatment from jute cellulose and also from the pith of *Aralia papyrifera*. The products obtained from these several sources we proved to have the same composition, *viz.*,  $\text{C}_{18}\text{H}_{26}\text{O}_{16}$ , differing from cellulose by  $-\text{H}_4 + \text{O}$ . This derivative resembles in many of its properties the original cellulose. Thus, when dissolved in concentrated sulphuric acid it is converted into a gummy substance, dextro-rotatory, and otherwise resembling ordinary dextrine. Treated with a mixture of nitric and sulphuric acid it is converted into the nitrated compound  $\text{C}_{18}\text{H}_{23}\text{O}_{16}3(\text{NO}_2)$ . The important feature of this decomposition of cellulose by nitric acid is undoubtedly the residual oxycellulose which we have just described, and the retention by this of the main chemical features of the original cellulose molecule, from which, as we have seen, it differs chiefly in manifesting feebly acid properties, and a considerable capacity for hydration (gelatinisation). At the same time that we examined this oxycellulose, produced in the laboratory, we noticed in our examination of certain natural fibres, the results of which we published in conjunction with Mr. Webster (*Chem. Soc. Jour.*, Jan., 1883), that some of these appeared to contain similar

products of the oxidation of cellulose. We also observed that these manifested a strong attraction for certain colouring matters, considerably in excess of that shown by lignified fibres under the same conditions, whereas, in the regard to the majority of colouring matters, the dyeing capacity of these latter fibres is much greater. We, in fact, proposed, on the basis of these results, a dyeing test of the presence of oxidised derivatives of cellulose. Almost simultaneously with the publication of these results there appeared in the *Journal of the Société Industrielle de Rouen*, a remarkable paper by M. G. Witz, a vice-president of that society, detailing the results of an exhaustive study of the oxidation of cellulose (cotton) by solutions of bleaching powder—from a point of view, therefore, of peculiar interest to technologists. We have recently had an opportunity of visiting M. Witz, and we have been so impressed with the importance of his researches, in their practical as well as theoretical bearings, that we think it desirable to lay before the Society an account of their main results, referring those who desire a more complete knowledge to the original papers in the *Bull. Soc. Ind. Rouen*, [10], 5, 416, and [11], 2, 169; also to *Dingl. Polyt. Jour.* 250, 271.

The bleaching operations in the works with which M. Witz is connected, came, a few years since, under suspicion, in consequence of a certain proportion of the finished pieces presenting the appearance of being riddled with a number of minute holes. On making specimen dyeings of these pieces with certain aniline colours it was found that the holes were picked out as dark spots upon a light ground, the result being due to a powerful attraction of the colour at the margin of the holes. It was then found that a result precisely similar was obtained by throwing a fine dust of bleaching powder upon ordinary bleached calico, previously wetted, and allowing it to stand some time, then washing off and dyeing. In this way the spoiling of the goods was traced to the injudicious manipulation of bleaching powder in the neighbourhood of the washing tanks. The cause of the accident was removed, but not until it had served the end of science in opening up a field for investigation. The next extension of these preliminary results was to throw light upon the cause of the familiar occurrence of brown patches in the whites of printed goods, the development of the brown colour always taking place during the steaming of the goods, and being accompanied by a "tendering" of the cloth. This effect it had been usual to ascribe to defective bleaching. It was now, however, found that the behaviour of the brown patches towards the aniline colours and other reagents was similar to that of cloth which had been oxidised by bleaching powder in the manner described above. It had also long been known that these patches were never visible till after the steaming. From these materials a deductive investigation was proposed, the result of which was to prove beyond doubt that the phenomenon was one not of deficient but of over oxidation. The immediate cause was then not far to seek. It lies in those circumstances which bring about an unequal action of the bleaching solution. The more prominent of these we will briefly elucidate.

1. *Action of the Air*.—To demonstrate the joint action of air and a solution of bleaching powder upon cotton M. Witz suspends vertically a strip of dry bleached calico about eight inches in length, with its lower end immersed to the distance of one inch in a solution of 4° B. At the expiration of one hour it is washed off and dyed in methylene blue—a solution of  $\frac{1}{2000}$  of the colouring matter in the cold—with the following result: The portion which has been immersed shows an even pale tint equal in tone to

that of the upper portion; the level of the liquid is sharply marked off by a deep shade of blue, which attains its maximum at a height of about one inch from the surface, the colour then shading off with perfect regularity into the upper zone which, not having absorbed the solution, represents the dyeing capacity of the original calico. Experiment then shows that this powerful oxidation of the cellulose is due, not to any direct action of the air, nor of its oxygen, but to that of the carbonic acid which it contains; and further, in the absence of a liquid surrounding, containing excess of the bleaching agent, to that resulting from the oxidation of the fibre constituents which together react upon the hypochlorite slowly but continuously absorbed. This powerful action of carbonic acid is easily demonstrated by the experiment of moistening two strips of calico with the bleaching solution (4° B.) and plunging them, the one into an atmosphere freed from  $\text{CO}_2$ , the other into an atmosphere vitiated by respiration. Ten minutes' exposure, followed by the dyeing process, reveals a pronounced difference between the two, the shade of blue being considerably stronger in the case of the latter.

2. *Action of Heat*.—Experiments with dilute solutions (0°·5 B.) showed that up to 60° no sensible difference is caused by rise of temperature. Above this point, the temperature being allowed to rise at the uniform rate of about 2° per minute, and the strips being removed at the several points named, the following effects were observed after dyeing in methylene blue: 65°, no sensible difference from the original, from that point increasing in dyeing capacity, until after exposure to a temperature of 75° a marked increase was observed; after 85° the dyeing reached a middle shade; after 90° a deep-blue was obtained, with a marked weakening of the tissue, these effects still more pronounced after 95°; at the boiling point the cotton was entirely resolved into a white powder, which, however, after dyeing, is seen to have less capacity for taking up the colour than that modified as described under (1). The elementary composition of the cellulose thus modified was found to be C 43·6, H 6·8. The substance was dried *in vacuo* at 100° previously to the analysis.

3. *Action of Light*.—When calico moistened with solutions of bleaching powder is exposed, with exclusion of air, for ten minutes to the direct rays of the sun, a marked increase in the oxidising action of the solution is observed.

4. *Subsidiary Influences*.—Amongst these, M. Witz has investigated the effect of keeping the bleaching solution in continual motion, and other minor points, from which, however, we must pass, with this brief notice, to consider his main conclusions, practical and theoretical. The action of solutions of bleaching powder upon cotton is not confined to the colouring and other matters which are oxidised and removed in the form of soluble derivatives, but extends to the cellulose itself. By the extreme action of the hypochlorite upon cellulose it is converted into an oxidised derivative, obtained as a friable powder, outwardly resembling starch. The analysis of the products obtained by the prolonged action in the cold, in the manner described under (1), gave the following percentage numbers—

Oxycellulose.				Cellulose, C <sub>6</sub> H <sub>10</sub> O <sub>5</sub>	
C	43·78	43·47	.....	C	44·4
H	5·85	6·13	.....	H	6·2
O	50·37	50·40	.....	O	49·4

the difference from cellulose being such as to suggest to M. Witz the term oxycellulose whereby to describe this substance. Considered in relation to the sub-



stance described by ourselves under the same name the resemblance is sufficiently close to warrant an identical term, the latter being the product of a more drastic oxidation, being indeed a more highly-oxidised derivative. We propose to distinguish it from the oxycellulose of M. Witz by the prefix  $\beta$ . At the same time we would guard ourselves against attaching an undue importance to the terms which we use in the embryo stages of our investigations. We do not assert that in our  $\beta$ -oxycellulose we have a final product of the action of nitric acid under the particular conditions of the experiment, nor can we prove that it is actually a single or homogeneous derivative. Attaching to it, therefore, rather a group significance, and conceding that we may have in the product described by us an aggregate of several  $\beta$ -oxycelluloses, the term thus qualified may be allowed to stand. In his study of  $\alpha$ -oxycellulose, M. Witz has observed that it is attacked by boiling alkaline solutions, a portion being converted into soluble derivatives of a deep yellow colour. For this reason he is inclined to reserve the term in question to the insoluble residue, regarding the action of the alkali as evidence of the presence of a second substance in the oxidised cotton, which from its reactions, viz., that described above, together with its reduction of an alkaline solution of copper oxide, appears to be closely allied to the glucoses. At present we offer no opinion upon this interpretation, as M. Witz is engaged in the further development of his views. The view which he takes of the modification of the cellulose molecule under the action of the oxidising agents is in general terms as follows: Cellulose offers two main points of attack, the first being such as to indicate the presence of alcoholic OH groups, the second a capacity for that mode of union with the elements of water (hydration) which is accompanied by an alteration of molecular structure, i.e., condensation and its reverse. The oxidation in question having no action upon the carbon nucleus, consists in a removal of H with a corresponding fixation of O, together with  $H_2O$ , the latter being probably attended by the structural modification already noticed. The general result of the action is the manifestation by the product of the properties of an *aldehyde*, and in a certain measure also those of an acid. This view has, of course, been arrived at only by a study of the action in extreme cases, but is none the less applicable to the whole subject of bleaching by oxidising agents, as indeed can easily be demonstrated. The development of these properties, the result of the actual structure of the cellulose molecule, affords a key to the whole practice of bleaching and printing, which needs, we feel, only to be suggested to be followed up by all who have an acquaintance with the fundamental truths of chemistry. We will, before closing this part of our subject, consider certain consequences of these results. In M. Witz's paper will be found a complete classification of all the better known soluble colouring matters, into those (1) attracted and (2) repelled by oxycellulose, of which we may select certain as typical—

(1)	(2)
Methylene blue	Diphenylamine blue
Hofmann violet	Induline blue
Malachite green	Tropaeolines
Safranine red	Eosines
Fuchsin red	Indigo carmine
Bismarck brown	
Phosphine yellow	

Generally, therefore, under (1) we find the basic colouring matters, under (2) acids and such substances as require the intervention of the ordinary mordants. The application of these observations to the diagnosis of the condition of a cellulose in regard to oxidation we have already indicated. The two blue colouring matters known as methylene and diphenyl-

amine blue are to be recommended for the purpose, the results obtained being complementary to one another. The dyeing is performed in weak solutions ( $\frac{1}{1000}$ ) in the cold; the time requisite is about twenty minutes, this being a convenient standard. If the substance to be examined has been bleached with chloride of lime the following process of purification must be carried out prior to the dyeing: After thorough washing (1), exposure to dilute nitric acid ( $2^\circ$  B.) for half an hour at the ordinary temperature; after washing (2), treatment with a boiling solution of sodium bisulphite ( $1^\circ$  B.) for 15 minutes; after washing (3), treatment with dilute hydrochloric acid ( $2^\circ$  B.) in the cold for half an hour; and lastly (4), prolonged washing with water. It is noteworthy that oxycellulose is extremely refractory towards all influences which might be calculated to reconvert it into cellulose. No reducing action hitherto employed has in the least succeeded in accomplishing this regeneration. In the method of dyeing above described, the practical bleacher will doubtless see a ready means of detecting faulty work. In the earlier experiments of M. Witz he will see indicated the following points to be observed in his operations: (1) To construct the bleach-house so as to admit only diffused light. (2) To keep the goods well submerged in the bleaching solutions. (3) To avoid undue exposure of the goods to the air in contact with the bleaching solutions. (4) To keep the air of the bleach-house as pure as possible, purity meaning freedom from acid impurities, in which, in addition to carbonic acid, we must include in certain cases the acetic acid which pervades the atmosphere of printworks. (5) To avoid the use of strong solutions of bleach, the limit recommended under the ordinary conditions of working being a solution marking  $0.5^\circ$  B. (6) In goods which are bleached for printing, especially in the case of steam styles, to arrest the process before attaining a good white, leaving it to the subsequent operations, all of which are oxidising in their action, to effect the discharge of the residual tint. Other minor points it is unnecessary to detail.

One of the most prominent of the results of M. Witz's investigations is, to use his own words, to show that a trace of carbonic acid has the effect of rendering a solution of bleaching powder infinitely more active as an oxidant; and it will be convenient here to notice a process of bleaching, recently patented in this and other countries by Mr. J. B. Thompson, since it is a complete adaptation of the points here elucidated to a system of economical bleaching. It has been sufficiently shown that the action of the bleaching solution is not confined to the non-cellulose constituents of the raw cotton, but considerably affects the cellulose itself. Now this subsidiary result will obviously be considerably lessened if to the non-cellulose constituents, which are by far the more easily oxidised, the oxidising agent be presented in minimum quantities. This follows from the simplest laws of the influence of mass upon chemical attraction. Mr. Thompson bleaches by a successive supply of such minimum quantities, at the same time employing an atmosphere of carbonic acid, under slight pressure, to decompose each additional quantity. The exact *modus operandi* consists in saturating the goods, previously scoured and introduced into a keir of special construction, with a very weak solution of bleaching powder, the excess of which is allowed to run off, and the goods being sufficiently drained the gas is admitted. These processes are repeated alternately until the bleaching is complete. The theoretical anticipations of the advantages of this process are fully confirmed by experience of working on the large scale. The economy in time and chemicals, and especially

in manipulation, has proved very great, and the entire cost of the process has been reported by a practical bleacher to be under 30s. a ton. We refrain from discussing the more practical bearings of this method, as it is our intention, favoured by the kindness of Mr. Thompson and those who are associated with him, to demonstrate, at the next meeting of the Society, the entire process.

We pass on to consider briefly the second portion of M. Witz's researches. The conversion of cellulose into oxycellulose is attended by a modification in the attractive power of the fibre for the various metallic substances used as mordants. We can only consider the most remarkable of these results, a result which has an altogether exceptional interest. The absorptive power of oxycellulose for vanadium exceeds anything yet brought to light in the nature of a qualitative reaction. Calico converted into oxycellulose by the method above described is capable of withdrawing the vanadium from a solution containing 10000000000 of the metal, and the combination can be demonstrated by printing the tissue with aniline black mixture! No one can fail to be struck by a reaction of such extraordinary delicacy, and many will be sufficiently so to acquaint themselves with the details of an investigation which has established so remarkable a fact. Of great interest also to the calico printer are the applications of this oxidation of cellulose as a substantive printing process. A large number of oxidising agents exert a similar action upon cotton in converting it into oxycellulose. It is only necessary to localise the action of them, by any of the familiar methods, so as to produce a design in oxycellulose, to obtain by a subsequent dyeing operation a corresponding result in colour. A simple demonstration of this action and the mode of utilising it consists in writing upon paper with an ink composed of a solution of potassium chlorate acidified with hydrochloric acid. After drying, the characters are indelibly impressed as oxycellulose, and may be brought out by soaking the paper in a solution of one of the dyestuffs previously mentioned. It is noteworthy that they may be bleached away any number of times by exposure to a bleaching solution, and may be made to reappear in any colour by treatment in a dye-bath. This remarkable stability of the oxycellulose supplies an additional caution to the bleacher of cellulose goods, since it further shows the impossibility of correcting the over oxidation of the tissue, the use of an antichlore for the same purpose having been shown to be unavailing, in consequence of the resistance of oxycellulose to the action of reducing agents. Oxycellulose is thus shown to be an unchangeable mordant, and the results obtained by M. Witz open out the prospect of realising to some extent the truly ideal method of fixing colours upon cellulose, namely, by so modifying it chemically as to enable it to combine with these directly. M. Witz has also studied the action of a large number of other oxidising agents upon cellulose. Ozone and hydrogen peroxide rapidly effect its conversion into oxycellulose. The degradation of cellulose fabrics by long exposure to the air is also due to the molecular change accompanying the formation of this product. Witz also applies his results to the solution of certain special points in the printing of calicoes. The use of violet to correct the greening of aniline blacks, as suggested by Brandt (*Dingl. Polyt. Jour.* 215, 453), was successful by reason of the formation of oxycellulose in the printed portion, this acting as a mordant in the dye-bath. The tendering of the cloth often observed in the printing of chrome yellows is referable to a conversion into oxycellulose. The question whether a pattern has been printed by a discharge or reserve may be pronounced upon by an

examination for oxycellulose. Lastly, Witz shows that the presence of a caustic alkali in oxidising solution prevents the formation of oxycellulose, its oxidising action being confined to the production of soluble derivatives. Thus an alkaline hypochlorite or an alkaline solution of potassium ferriyanide may be used to effect oxidation in their several degrees when the formation of oxycellulose is especially to be avoided. Without entering into details, which would only be of special interest to those who make a close study of these subjects, we would point out, as a matter of general scientific interest, that the parallelism between the investigations of M. Witz on cellulose and of ourselves upon jute, and the compound celluloses of which it is a type, has been of the closest nature. Not only have they been coincident in time, but the point of departure was similar in the two cases, viz., the disintegration and discolouration of bleached fabrics, as a result of an oxidation in the bleaching process, further developed in the process of steaming. Their point of contact lay in the investigation of the two forms of oxycellulose above described. On the occasion of our visit to M. Witz we made with him a study of the cellulose isolated from jute by the method of chlorination. This has been shown to differ in composition from ordinary cellulose, the analytical numbers obtained by us showing a close resemblance to those obtained by M. Rosenstichl for  $\alpha$ -oxycellulose.

Cellulose from Jute.				$\alpha$ -Oxycellulose.	
C	43.2	43.5	43.0	43.62	
H	6.2	6.0	5.9	6.00	

In confirmation of these results we found a close similarity in properties, *e.g.*, in tinctorial capacity and in the power of reducing an alkaline solution of copper oxide. Jute cellulose appears therefore to be oxycellulose. The same is doubtless true of a large number of the so-called celluloses used in paper-making, which are isolated by chemical treatment from compound celluloses, and M. Witz's results will doubtless prove to have an important bearing upon this branch of the cellulose industry.

In conclusion, we will briefly describe the results of the preliminary study of the oxidation of cellulose by means of a solution of potassium permanganate, to which sufficient caustic alkali was added from time to time to prevent the solution from becoming acid. The cellulose was employed in the form in which it is precipitated from its solution in the Schweitzer reagent, by the addition of acid. After washing, this was suspended in the solution of permanganate, of which further quantities were successively added until the whole of the cellulose was converted into a soluble derivative. This was then isolated by means of its lead compound, being purified by fractional precipitation, and thoroughly washing the precipitate. On decomposing this with hydrogen sulphide, filtering from lead sulphide, and evaporating, a strongly acid gummy body was obtained, which dried at 110-120° to a yellow friable solid. This was analysed with the following results: 0.2204 (deducting ash) gave 0.3038  $\times$  0.004 ( $\text{CO}_2$  determined in the ash) and 0.0622  $\text{C}_2\text{O}$ .

Calc. $\text{C}_{12}\text{H}_{10}\text{O}_{12}$			
C	....	38.08	....
H	....	3.13	....
			38.10
			3.01

The resemblance of this body to Fremy's metapectic acid is very marked. The entire series of bodies of which metapectic acid is the extreme member are obviously oxidised derivatives of cellulose; and in the two methods of oxidising cellulose which we have studied, viz., by nitric acid and by alkaline perman-



ganate, we have arrived at products presenting a close relationship to bodies of the pectic series. We are prosecuting these researches in the hope of throwing some light upon an important group of natural products which as yet remain without the pale of chemical classification. This brings us to the conclusion of our subject. We have only to hope that it will contribute in some measure to the building up of the fabric of cellulose technology, by suggesting directions in which work is so urgently needed.

The discussion was adjourned to the meeting in May, when Mr. Cross will demonstrate the processes referred to in the paper.

After the reading of the above paper a conference was held on

#### STANDARD METHODS OF SAMPLING MERCHANTABLE PRODUCTS, OF ANALYSING THEM, AND STATING THE RESULTS.

The CHAIRMAN: We have to discuss this evening, as the circular which has been sent to you indicates, the desirability and practicability of arriving at a national or international agreement concerning the methods of sampling, the methods of analysis, and the modes of returning the results of ores, raw products, and chemicals. This important subject has already been discussed by the Liverpool, London, and Newcastle Sections of our Society, and we have thus the great advantage of knowing the views of many eminent chemists, and of men well qualified to express an opinion on it. There appears to be no doubt in the minds of most of the gentlemen who took part in these discussions that such an agreement would be very desirable, and this needs not to surprise us. We all know that there exist in many instances various methods of analysis for the same substance, and we all equally well know that the results of an analysis of the identical substance, but obtained by different methods, may lead to different results. It does not therefore require much consideration in order to come to the conclusion, providing that such an agreement is practicable, that it would be a most desirable one, and I think that it will be superfluous to discuss this evening the question of desirability. The difficulties which present themselves to your minds, and the means of overcoming these difficulties in order to come to a satisfactory agreement, ought to constitute the principal object of our discussion. We may perhaps also dispense this evening with the enumeration of the great differences and discrepancies which no doubt many of you have met with in your various experiences, and which resulted either from the different methods of taking samples or from the different methods of analysis or from other causes. However interesting these communications might be, I think that we may fully accept as an established fact that the present system has its great disadvantages. I propose, in order not to waste your valuable time, that we consider *seriatim* the following questions, which I beg now to submit to you, and after having heard your views, that we then bring forward at the next meeting resolutions embodying your replies and opinions. I only desire to make one exception, as I intend, with your permission, to bring later on a resolution before you in reference to the first question. (1) Is an international or a national agreement more feasible? (2) Does a national agreement meet all purposes, at least

as far as the interests of this country are concerned? I beg you to carefully consider these two questions, as there may be some insurmountable difficulties of arriving at an international agreement, while the less ambitious scheme of arriving at a national understanding might be far more practicable and equally as useful. (3) What are your opinions concerning uniform methods of sampling, and how could an agreement be arrived at? (4) What are your opinions concerning uniform methods of analysis? (5) What are your opinions concerning uniform methods of returning the results? (6) In the event of the appointment of a representative committee, (a) how should that committee be constituted? (b) Should it be a permanent one? (c) What organ do you consider the most serviceable to give effect to the conclusions to which that committee might arrive? (7) If standard methods have been agreed upon, and have been published by the authority of that committee, should these be considered binding to either chemists, buyers, sellers, or consumers, or should they be only considered as recommendations? However successful we may be in coming to some agreement, it would be in my mind not alone not advisable, but also almost impossible to make any agreement in any way binding on chemists, buyers, sellers, and consumers. Such an agreement, of whatever nature it might be, ought to be only considered as a recommendation to the chemical profession or others, but I do think that any recommendation made by a representative committee would be in most cases adopted. I have now the pleasure of calling upon Mr. George E. Davis to state his views upon the subject.

Mr. GEORGE E. DAVIS: I think, Mr. Chairman, that there are very few present who will disagree with you in your remarks that an international method or methods for sampling goods, for their analyses or assay, relating also to the method of returning the results, would be a very desirable thing; but in each of the discussions held at those Sections where this subject has been considered the means whereby this most desirable end could be accomplished has been avoided. No doubt it is a most difficult question to approach, and I am not sure whether every difficulty could not be met by attention to the methods of sampling and the modes of returning the results of the analysis or assay, leaving the analytical chemists still responsible for discrepancies if they choose to perform inaccurate work by incorrect methods. An enormous amount of abuse has been poured upon the heads of chemists on account of discrepancies, which without further inquiry would make the outside public believe that chemistry was not a very exact science. But may not much of this be due to the method of sampling? My experience in works of very varied character leads me to positively assert that it is in many cases the sampling which is the inexact process, and when several chemists have been given samples, in which every care has been taken to render them homogeneous, their results have not been very far from each other, in most cases certainly within the limits of error of experiment. It would, in my opinion, be a step in the right direction, in the case of samples to be sent to more than one chemist, if instructions as to the methods of sampling were issued. It should be made a point that the contents of the several bottles be well mixed, ground up either in a mill or in a mortar, passed through a sieve, and the homogeneous mixture rebottled. Of course there are substances which could not be treated in this way. Substances which are deliquescent or hygroscopic would need special treatment, as would also pasty substances, but this is a matter of detail which would have to be worked

out, the main point I would wish to insure being that when samples were to be examined by different chemists, possibly one acting for the buyer and another for the seller, it should be an undeniable fact that both were working upon identically the same substances. Of course, one can never expect to be able to cope with what I may term fraudulent sampling, if the sampler, say in sampling burned pyrites, peppers the sample with a little copper precipitate, or if in sampling a weak manganese ore he manages to insert a tolerable quantity of good 80 per cent. into the bottle, or in sampling bleach he contrives to save the lumps and lose the dry dust, the fault does not lie with the analyst, and the injured party has his remedy at common law. An agreement upon the methods of returning the results of analysis would be most beneficial, and in my opinion be one way out of the difficulty of forcing standard methods of analysis upon unwilling minds. Take, for instance, the question raised at Liverpool by the chairman of that Section regarding the estimation of the available peroxide of manganese in manganese ores of commerce. He says, "We found it desirable to use the protoxide of iron test," which no doubt, though in every respect at that date admirably suited to the valuation of its chlorine producing power is now nevertheless a little misleading, and a test which does not accurately represent its value even to the chlorine producer, while for such purposes as the manufacture of spiegel or ferro-manganese the method used by the alkali makers could not be entertained at all. In fact, I know of a case in Manchester where a manufacturer bought a parcel of "manganese" by the usual test, but the seller, having found that it would be used for sulphate of manganese making, had it analysed for total manganese present, and this departure led to no small dispute between the two parties. This could have been prevented, even with our present methods of sampling, by an agreement as to the best methods of stating the results. It is in cases of this kind that the difficulties of insisting upon standard methods come in, as the seller as well as the buyer has to be protected, and in the methods already quoted there would have to be a standard method for the use of chlorine producers, another method for the use of ferro-manganese makers, and another for the maker of sulphate of manganese, where the maximum of protoxide or carbonate is an advantage. Surely chemists may be left to estimate the active principles of these substances for the various purposes of trade, if the particular trade in question will only say how it desires the results expressed, but it is obvious that owing to the impure and varying nature of all minerals and raw materials, one could not depend upon a factor for converting the results of one standard method into the probable results which might be obtained by another. Acetate of lime is a substance which has caused much dispute. Some chemists estimate the amount of acetic acid in it by distilling with phosphoric acid, while others use a method known as the sulphate of soda method, giving results some 8 to 10 per cent. higher than the former. Of course buyers and sellers know the £ s. d. difference between the value per unit of these tests, but the question may be fairly asked whether a chemist is doing right by certifying that a sample contains a certain percentage of acetic acid when he knows full well that it does not. The analysis of manures, potash salts, all raw products, and finished articles, may, in my opinion, be all dealt with in the same way, viz., by an agreement as to the method of stating results. My views are not at all favourable to the standardisation of methods, without such methods are founded upon absolute scientific reason-

ing and experiment. Let me explain by calling attention to the testing of crude benzol, mentioned by Mr. Watson Smith, at the Liverpool meeting. Quite recently I had occasion to send several samples of crude benzol to various chemists, and by the ordinary test they all returned the certificates within a very few points of each other, but they also mentioned that the sample was an abnormal one, and that they had modified the "usual test," and by this modification, I am glad to tell you, they were again within very few points of each other. This circumstance leads me to suppose that chemists are capable of taking care of their reputation, and that there is but little need for fixing standard methods, at least for many substances. One thing I would earnestly like to see abolished, and that is the host of ammonia-meters, aerometers, Twaddell's and Baumé's hydrometers, the old Fahrenheit thermometer, and all barbarisms of this kind, and to place in their stead the ordinary specific gravity glass, taking water at 1000 as the basis. I think this might be done by publishing a bold table of the equivalents of the various hydrometers in specific gravities, in the Journal of the Society, and continuing to publish and exhibit the same on the cover of the Journal for months, or even years if necessary. In conclusion, I think national methods for accomplishing the above objects are attainable; but not international ones.

Mr. MELLOR: I am not prepared to endorse the suggestion that our Society should recommend the adoption of standard methods of analysis. Chemistry is so vast a science, consisting as it does of almost an infinite variety of truths or facts, that it admits of the attainment of the same results, accuracy of analysis, by many and varied processes. Chemistry is a growing science, and to limit it, to confine it, to chain it down to the prescribed limits of "standard methods of analysis," would most assuredly interfere with the free growth and development of the science that we all love so well. But I think that our Society could well adopt the recommendation of a more perfect and therefore uniform system of sampling than that which now prevails. From some experience I am prepared to affirm that difference of analysis is more the result of imperfect sampling than of inability in the analyst or of errors in the methods of analysis. In proof of this I may state a recent experience. A cargo of mineral arrived in the Mersey a few months ago. A sample of it submitted to the buyer gave 29 per cent. of metal—the certificate of it from a foreign chemist showed it to contain 33 per cent. of metal—but when the whole cargo arrived at its destination, and it had been properly ground and sampled, the whole bulk contained 40 per cent. of metal. All the analyses in this case were correct—it was the sampling that was at fault. Simply as suggestive, and as my contribution to this conference, I name the method of sampling minerals as adopted at Patricroft. On the arrival of the mineral it is weighed, and every tenth portion—whether of bags, or casks, or other packages, is laid aside. This tenth part is afterwards ground, mixed with a spade on the floor, and then passed through a rather coarse sieve to insure its more complete mixing, and then the foreman takes an iron tube, about 4ft. long and some 1½ in. or 2 in. diameter, and probes the heap of ground mineral at top, bottom, sides—in a word, all over it. The sample thus obtained is then divided into three portions—one for the buyers, one for the sellers, and one for reference in case of dispute—and all carefully sealed up. One source of error is the question of moisture, and the mode that it is to be dealt with ought always to be agreed upon between buyers and sellers, and the analyst ought to be informed of the arrangement



entered into. Must the sample be dried in a water-bath for twelve hours at 100° C., or is the chemist to dry the sample in his own way, and in his own time, and in an open dish over an open furnace? Clearly much difference of results would be obtained if clear and definite instructions on these points were given to the analytical chemist. Another source of error, and the fruitful cause of difference of results obtained by chemists, arises from the loose and indefinite way in which the words "assay" and "analysis" are so frequently employed. Buyers and sellers, and the chemists acting for each, ought to know each other's minds clearly on this matter, and in all agreements between buyer and seller and instructions given to chemists the words, contents "fire assay," or contents by "analysis by the wet or acid process," ought to be introduced. It is only necessary to point out that in all cases of volatile metals, of which antimony and bismuth may be taken as types, even in the most skilful hands the difference in the results obtained between "fire assay" and "analysis by acid process" is from 2 to 3 per cent. of metal contents. I fear that the question of an international agreement as to the adoption of standard methods of analysis and of sampling is at present not practicable, the conversion of kilos. and metres into pounds and inches of itself presenting a grave difficulty, but I think that it is within the power and scope of our Society to recommend the adoption of some uniform and standard method of sampling for the United Kingdom; and if we succeed in this, we shall be justified in inviting other nations to join us. Let us aim at realising the attainable.

Professor ROSCOE wrote—

"In consequence of the meeting of the Literary and Philosophical Society, Professor Roscoe regrets that he cannot be present at the sectional discussion on methods of analysis. He wishes, however, through Mr. Watson Smith, to express his opinion as to the very great importance of the question, and the desirability of the full discussion. He feels unable to make any remarks on the advisability of fixing definite rules for sampling, but it is clear that correct sampling is an all-important factor, and that if some well-defined methods can be laid down and generally adopted the result must be of value. As regards the further question of the Society laying down the law as to methods of analysis, he sees much greater difficulty, and he thinks that this, if done at all, should only be done after very careful consideration. What is really needed is an appreciation, both by the analyst as well as by the buyer and seller, of good and correct methods. Until the public as well as analyst are well educated in this respect there will always be a demand for uncertain or even inaccurate methods, some of which are still in vogue. To fix upon certain processes is to do away with the freedom and discretion of the analyst, and possibly to introduce the very evils it is desired to avoid, inasmuch as new and improved methods are constantly being introduced into science which a stereotyped system would find difficult of adoption. Hence Dr. Roscoe is inclined to believe that the most advisable course for the Society to take is rather to point out those methods which are faulty, and suggest others which are free from errors, than to lay down any hard and fast lines with regard to analytical methods. He would also remind the Section that the Institute of Chemistry has for its object the attainment of the same end, viz., the reliability and accuracy of commercial analyses, by a somewhat different means, viz., the certifying of competency on the part of the analyst, by examination or otherwise. Dr. Roscoe feels that, after all, the best way to secure a good analysis is to employ a good man to do the work, and he would rather trust to his discretion and skill in choice of methods than to the less educated person working by rule."

MR. WATSON SMITH: Since speaking in Liverpool and writing to London on the subject of standard methods of analysis and sampling, I have thought more on the subject, and my earlier views, never very clearly formed, and therefore never very clearly stated, as to the possibility and desirability of the general adoption of such standard methods, have received considerable modification. I confess I have now almost come to the conclusion that, on the whole, we may perhaps do best to leave the analytical methods to look after themselves—to commend or condemn themselves, as the case may be, in the belief that the Darwinian law of the "survival of the fittest"

will also extend its truth to them. If it be said that as improved methods appear those methods employed in the standard code could be replaced or altered, I would reply, "Not without much delay and great difficulty, for according as the alterations involved would for the time affect manufacturer or purchaser, seller or buyer, so would the one or other class contest the innovation." But in the proposed international scheme one analytical method was to be fixed upon for each kind of quantitative determination, and all the chosen methods were to become obligatory as regards commercial products. As regards this kind of compulsion another view presents itself, and not a pleasing one, of analysts of commercial products treated like animated testing-machines, worked by a select committee. However, do what we will, follow what course we please, place ourselves under the control and guidance of whatever select committee of chemists we like, we cannot cause to be infused a single ray of life or intelligence into analytical methods themselves, nor can we teach a test-tube, a chemical balance, or a burette how to think! It is to be hoped, though, that with the increasing educational advantages and means offering, and likely to offer yet more abundantly in this country, that chemists in plenty will be found, both sufficiently instructed and experienced to discriminate between the reliable and unreliable in analytical methods, and with minds sufficiently enlightened to choose the good rather than the bad. The Institute of Chemistry deserves to be upheld and supported more generally than it is, for it does a good, albeit a quiet work, in practically discriminating between those young men who have received an education fitting them to become efficient analysts and those who have not, and in doing this adopts a very safe, because a very high, standard in its examinations for the associateship. If a buyer or seller wants a reliable analyst, let him run his eye over the list of the Institute and select there, if he be at a loss. Once more returning to the compulsory scheme, and now referring to the proposed select committee, whose office it would be to choose the methods and issue them, it seems to me the time has scarcely come yet for us to subject ourselves to what might develop into something analogous to a kind of chemical priestcraft! I think, in short, the attempt through the medium of a select committee, or otherwise, to impose the adoption of a list of compulsory analytical methods on chemists of education and experience would appear to the latter very objectionable. I can both understand and appreciate the publication and recommendation in the columns of our Journal, or of any other journal, of serviceable and accurate methods, and also the exposure of unreliable and deceptive methods, but do not let us, as it were, take the intelligence, honour skill, and understanding of the chemist from him and place them in his standard solutions, his burettes, and his evaporating dishes! The value of sets of rules for sampling various chemical products would be very great. Of special advantage would they be for those whose experience as regards outward appearances of such products is not yet sufficiently formed to enable them to guard themselves against the tricks that are available to professional samplers, making such samplers, in the hands and employ of, say, unscrupulous floaters of companies for exploiting minerals or mineral deposits, extremely dangerous persons. It is astonishing what some of these mineral samplers can do in the way of picking samples from one and the same heap. But even to some masters and proprietors such rules would be of great value (and to this I can testify from my own observation) to protect them from the wiles of certain foremen and



managers. I may ask, what is the use of employing a chemist at all in cases of such a kind, for he may only certify the strength of an unfair specimen. If any chemist wants to try an instructive experiment in this line, let him go over a heap of rather badly-made saltcakes from the ordinary pot and furnace, and take first a sample from every part with a spoon, and secondly another sample with a spade (i.e. large lumps with small), and test both separately, and he will find considerable difference in the percentages of sulphate discovered in the two specimens.

Mr. J. CARTER-BELL: This subject of standards has been for a long time in my thoughts. Many years since I published in the *Chemical News* a letter calling attention to some very inaccurate analyses of water, which water had been analysed by five different chemists, of whom no two agreed in their analyses. I then suggested that some standard methods should be employed. It is of no use, Mr. Chairman, to bring before you instances of incorrect analyses, as their name is legion. What one ought to do, is to make some suggestions and to give some sensible practical remarks. The question of sampling has been so ably taken up by Mr. George E. Davis and Mr. Mellor that it leaves nothing for me to say upon that point. It is unnecessary for me to state that I heartily agree with all that has been suggested by these gentlemen. Mr. Davis says that he does not think that the analytical chemists are at fault. It is very kind of him to speak a good word for a much-abused body of men, expected to be immaculate regarding the analyses performed. I do not agree with Mr. Davis on this particular subject. After an experience of twenty years' analytical practice, I do not find that "chemists in their little nests agree." My strong opinion is that we do want some recognised authority that should be able to give to chemists some standard methods of analysing commercial products. I think this meeting might learn a lesson from the Society of Public Analysts, who at this present time have formed a committee for the purpose of ascertaining the best method of analysing milk, for great variations occur even in the analysis of so simple a thing, for one man bases his conclusions upon weighing the solids, not fat, another weighs the fat, while a third weighs both. To settle which is the best method a committee of thirteen chemists has been formed, a sample of pure milk and one of adulterated milk are sent to each of the thirteen chemists, the analyses are made by each chemist according to his own particular plan. As the committee is still sitting it would be premature to state what will be the results. I cannot see why this Society, the Chemical Society, and the Institute of Chemistry should not form an influential committee to thoroughly investigate this question of analysis. To save time I will only give one illustration, and that shall be the analysis of sulphur pyrites. It is well known that chemists differ in the estimation of the sulphur to as much sometimes as 2 per cent. I think the chemist cannot complain and say that he does not receive the sample in a finely-powdered condition, so that there can be no excuse on the question of sample. The difference arises in the analysis from one chemist using the acid method while another uses the fusion process, and the result is the analyses cannot agree. Supposing a committee were formed, and a sample of two or three pounds of finely-ground sulphur ore obtained, which should be most accurately analysed, and every element in it determined. The ore should then be put into thirteen or fourteen bottles, and sent to as many chemists, with a request that they should estimate the moisture and the sulphur by their own processes. If when the returns were sent in it was found that a

large majority were close to the standard, and that they had all used a similar process, I think then the committee might safely say we recommend this process. The method should be published, and written out in the most minute detail, so that a chemist, if he had never performed such an analysis before, on reading over the method of the committee, would have no difficulty in returning correct results. To me it seems childish to say that the chemist will become a mere machine. If he is told what to do, and how he is to do it, instead of being a machine the chemist would have more time to devote to other work instead of wasting his time upon what is already known.

Dr. WATSON: There are some people who appear to imagine that it is possible to give complete instruction in every matter relating to the arts and manufactures by means of treatises or textbooks. My experience leads me to an opposite conclusion. I imagine most of you will agree with me that the knotty points that one meets with in one's own business are generally most skilfully avoided in such treatises, or infallible guides, and I am afraid no number of conferences and committee meetings would suffice to supply the expectations of those who would answer all the queries put to the meeting in the positive. Nevertheless the discussion of them cannot fail to do good apart from any particular action to be taken by our Society or our Section of it, by forcing on the attention of those who buy and sell raw materials and manufactured articles the importance of paying increased attention to that part of their contracts that relate to sample and assay. In the abstract it would appear as essential that a form of contract for raw materials or chemicals should define the method of sampling and assay as it is that it should fix the price or the other terms. Practically, this is met by there being in most cases a custom of the trade in sampling, and by the existence of faith in the power and willingness of the analyst to do what he undertakes to do. Those who have set in motion the discussion of these questions of standard methods tell us that the custom of the trade as regards sampling many articles is not a proper one, or that both a proper and an improper one exist, and further that some analysts employ methods of analysis or assay that are not to be depended upon, and thus they explain the discrepancies that occur between the results of the examination of the same parcel of certain commodities by different analysts. I daresay that is so, but I am sure also that other elements in these discrepancies are the idleness and carelessness on the part of samplers, and ignorance and incompetence, and what I had better perhaps call *bias* on the part of the analyst. No textbook instruction will suffice to eradicate these. With regard to most of the heavy products and raw materials of industry proper methods for both sampling and analysis are well understood, and do not remain to be discovered, although it is doubtless true that these proper methods are not universally employed. Allowing that it is desirable that all the transactions in a particular commodity should be conducted with reference to one standard method, and I am certainly of that opinion, I cannot see myself that it is possible for our Society to do more, at the most, than offer suggestions to the trades concerned. We can discuss, promote investigation, and suggest improvements, but after that we must leave it to those commercially concerned to adopt our suggestions. Our Society, by its constitution, is admirably adapted to do this. For example, suppose after we had heard Mr. Carter-Bell's admirable paper on this very subject of standard methods, where he entered into detail with regard to analysis and valuation of gas liquors, and it had been published, we



had had a meeting called specially to discuss the proposals in that paper, I doubt not that a number of persons interested would have attended, and we should have had a discussion among them that would probably have so threshed out the subject as to supply what might be adopted as a standard method for the valuation of gas liquors. As a practical way of promoting these objects I suggest the following as a feasible scheme. The general council should draw up a scheme allotting the discussion of the subject that relates to special raw materials or chemicals to each Section, so as to divide the labour. Pyrites to one, bleach to another, alkali to a third, and so on. By this division of labour time would be saved. The sectional committee would then arrange for the most thorough ventilation and discussion of the whole subject allotted to it, and afterwards draw up a report, and if possible definite regulations such as might form a standard for the trade. Our Journal would, in the ordinary course, publish reports of these meetings and the formal result of the discussion. So far I believe our Society can go. It can by its representatives come to definite conclusions and publish them, but it cannot give them the force of law. Even though these standards were not authoritatively adopted by the trade, they could and would probably be stipulated at first by buyers, who were perhaps not quite able to form an independent judgment, as a system in which they could have confidence, and be easily referred to as the Society of Chemical Industry's method. Taking the questions in our circular *seriatim* I would say (1) that I believe that when an article, uniform in its physical condition, is a large article of commerce, it is both practicable and desirable that a general agreement should be attained with regard to the way in which samples are taken, but I doubt if such a general agreement can be attained by any efforts of our Society. (2) No analyst ought to issue a certificate of analysis unless he is satisfied that a proper procedure has been followed. If he issues a certificate in the contrary belief he does a dishonest thing. There are two ways of diminishing dishonest practices. The first is by precept and example; the second by pains and penalties. Our part is evidently the former. If we do the work well, exposing erroneous practices, and providing for the analyst methods that have the approval and recommendation of our Society, I think that analysts will find it safer and more comfortable to avail themselves of these methods than to employ others, perhaps equally accurate, but which have not such approved recommendation. In this way, I believe a pretty general agreement may be arrived at. Further, I can see no reason why such a general agreement should not become an international one, but it would become so by operation beyond the scope of our Society. (3) This question implies that there are in some cases variations in the methods of returning results. If the several methods involve nothing contrary to truth they may be all admissible, but I think our Society should draw attention to variations of this kind calculated to deceive, such as the well-known case of the so-called Liverpool custom, in the alkali trade, and set its face against them. (4) As I have said before, I believe that our general council and the sectional committees are in a position to do all that we as a Society can do, and that to appoint special committees for the purposes of regulating the course of trade in this respect would be to go beyond the legitimate functions of the Society of Chemical Industry.

Mr. GRIMSHAW : In the first place I may say that my own view inclines very much in the direction pointed out by Mr. Davis and Mr. Mellor. I think with Mr. Bell in one respect that the formation of a committee to decide upon accurate methods of

analysis would be a very useful institution, but I do not think that the duty of that committee should further proceed in the way of asserting that analyses by one or even two particular methods should be adopted, as I am of opinion that no committee would be able to carry such methods into law. The great difficulty, to my mind, which has stood in the way of uniformity in the methods of analysis is simply the fact that there are "buyers" and there are "sellers." A buyer will always choose a method of analysis which gives a low valuation, and a seller will choose a method which gives a high one, a very good illustration of which you will find in relation to the "soda test," mention of which was made in the discussion at Liverpool. The Liverpool chemists appear to adhere to the old atomic weight of sodium, the consequence of which is that there is actually a difference in the results between Liverpool and Newcastle simply because in the one place a different atomic weight is adopted from the one in use at the other. Yet even this need not lead to any confusion between the buyer and seller, any more than a difference in the result given by different processes, if it is known by what standard the sample has been analysed, because, this being so, the value of the article can be assessed *pro rata*, according as the numerical result is known to be high or low, under those particular conditions. Leading from this point, we must not forget that in deciding on the method of analysis a chemist is very often biased in favour of a particular method, and it will always be the case that an analyst who has a great deal of work on his hands, will as far as he is able choose the most simple operation of those in use, and it seems to me that in many cases if that operation is one which does not give quite so theoretically correct a result as some other, yet it may be fairly considered sufficient for the purpose, because if it is a process which will give uniform results on its own basis, it need not attain to a superlative pitch of scientific accuracy so long as by its aid we are able to form an equitable judgment upon the results. The valuation of crude acetate of lime is a case in point. The sulphate of soda process may not give so accurate a measure of the real percentage of acetic acid as the distillation process, when very carefully conducted, though I do not think the difference is great, as is sometimes asserted, but it is much more easy and rapid of execution, and gives very concordant results, and we therefore cannot wonder that the small amount of time and attention which the sulphate of soda process demands compared with any process of distillation, has recommended the former to analysts who have a living to obtain. At the same time it is very desirable that a reliable comparison of these two methods of valuing this important chemical product should be made. I am myself working on this subject, and hope at some future day to be able to place my results before the Society. I practically agree with Mr. Davis that if we could only secure "good sampling" we should really be in a fair way to overcome all our difficulties, but in sampling there is, of course, a bias, and that you cannot eliminate by any method of legislation or the formation of committees. If methods of sampling were, however, taken under consideration, I think it would certainly come fully within the province of our committee to recommend for adoption certain modes of procedure for obtaining accurate samples of all the more important materials known to industrial chemistry.

The CHAIRMAN : I am glad to hear that Dr. Roscoe and Mr. Watson Smith agree with me, in case an agreement is practicable, that the same ought not to be a compulsory one. After the many valuable remarks we have heard, we may fairly conclude that

an international agreement would not be agreeable, but that a national one would be far more feasible. I therefore beg to move the following resolution: "That in the opinion of this meeting it would be practicable and very desirable to attain a national agreement on standard methods of sampling ores, raw products, and chemicals, but that an international agreement would be at present impracticable."

The resolution, which was seconded by Mr. DAVIS, was put to the meeting and carried unanimously.

The conference was then adjourned until May 6th.

## Glasgow and West of Scotland Section.

Chairman: Ed. C. C. Stanford.

Vice-chairman: Professor Ferguson.

### Committee:

George Beilby.	Professor Mills.
Professor Crum Brown.	James Napier, jun.
J. Christie.	T. L. Patterson.
Dr. John Clark.	J. B. Readman.
Professor Dittmar.	Dr. Edmund Ronalds.
George G. Henderson.	F. J. Rowan.
George M'Roberts	R. R. Tatlock.
James Mactear.	Dr. William Wallace.

Hon. Treasurer:

J. J. Coleman, 45, West Nile Street, Glasgow.

Hon. Secretary:

Dr. James J. Dobbie, Chemical Laboratory, University of Glasgow.

### MEETINGS FOR 1884.

MAY 13TH.—(1) On Different Methods of Producing Cold Artificially. By Mr. Coleman. (2) On the Composition of Tobacco. By Dr. Clark.

Meeting held Tuesday Evening, March 11, 1884.

MR. ED. C. C. STANFORD IN THE CHAIR.

THE CHAIRMAN intimated that the Section now numbered 137 members. When it was formed there were only 114 members of the Society resident in Scotland. Sixty-six new members had joined during the month that the Section had been in existence, bringing up the number of Scotch members of the Society to 180.

## ON THE EFFECT OF TEMPERATURE IN DYEING.

BY EDMUND J. MILLS AND A. G. RENNIE.

VERY few measurements have been made of the influence of heat in chemical change. Hood (*Phil. Mag.*, Nov. 1878), calculating from data previously published by Harcourt and Esson, was of opinion that chemical effect is proportional to the square of the temperature; and this result was afterwards confirmed by Warder. Our own object has been to make some preliminary measurements of the effect of temperature in dyeing. For this purpose we selected purified cashmere wool (in the piece) and a well-crystallised sample of rosaniline acetate. The first series of

experiments was performed in such a manner as only to saturate the wool at successively higher temperatures. To this end we took a glass bath holding in every case 200 cubic centimetres of water, heated to and kept at the desired temperature within a few hundredths of  $\pm 1^\circ \text{C}$ . A standard solution of the acetate was prepared, containing 0.1 grm. in a litre of water. The pieces of wool to be dyed averaged about 0.5 grm. each, and contained 12.73 per cent. of water. These were first introduced into hot water, in order to prevent the formation of air bubbles, drained rapidly, and inserted in the hot experimental bath, in which they were kept each for one hour. During this time rosaniline solution was slowly added, with constant stirring, until at the end of the specified time a red tint was just perceptible at the edge of the liquid. In some cases a little water of the same temperature as the bath was added to make up for a slight evaporation. The following table contains our numerical results, and a corresponding set of numbers calculated from the data marked with an asterisk, by means of an equation referred to below:—

Colour absorbed per gramme.	Cubic Centimetres.	Temperature.
Cubic centimetres.	Calc.	C.
—	0.00	—1.46
0.41	0.37	+1.50
0.90	0.90	6.25*
1.25	1.75	16.25
2.18	2.18	26.25*
2.42	2.18	36.25
2.21	1.82	46.25
1.21	1.21	56.25*
0.52	0.56	66.25
0.22	0.22	86.25*

It is already known (Mills and Thomson, *Chemical Society's Journal* 35, 28) that at some temperature approaching the boiling point of water, and if adequate time be given, rosaniline salts undergo dissociation, producing, if their solutions be very weak, completely colourless liquids. It is in fact evident, as well from the numbers above given as from the dyed samples themselves (exhibited to the Section), that throughout a considerable range of higher temperature the tendency of wool to absorb colour is more or less counteracted by a dissociation stress, though in our own experiments the time was too short for much actual dissociation.

If we assume Hood's law, and consider the absorption of colour as well as the dissociation stress to be chemical effects, the combined effect in these experiments should be proportional to the fourth power of the temperature. As the temperatures from 6.25 to 66.25 were taken at equidistant intervals, it is easy to examine the differences of rosaniline solution thereto corresponding. The sum of the fifth differences proves to be  $-0.7$ , or very nearly zero—a criterion of a quadric curve. The equation of this curve is—

$$y = b(t + 1.46) - c(t + 1.46)^2 - d(t + 1.46)^3 + e(t + 1.46)^4$$

where  $y$  is the amount of colour absorbed in cubic centimetres,  $t$  is the temperature, and  $b, c, d, e$  are constants of condition. The logarithms of these constants are—

log. $b = 1.1068445$	log. $d = 5.4194377$
log. $c = 3.1009246$	log. $e = 7.4435168$

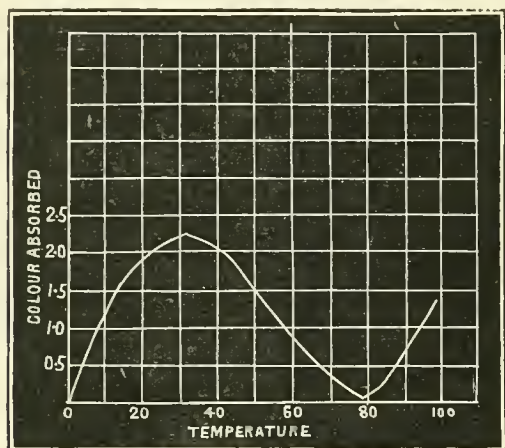
Hence we have calculated the position of the following points of interest, viz:—

Temperature C.	Effect.
—1.46	No colour deposited.
+31.11	Most colour deposited.
81.15	Very little colour (0.7 cubic centimetre) deposited.

Beyond this last temperature more colour is gradually taken up, the calculated amount being 1.30 cubic centimetres at  $100^\circ \text{C}$ ., so that the advantage still lies



greatly with the temperature  $31.11^{\circ}\text{C}$ . At this point 1 grm. wool is dyed a fairly deep shade by 2.23 cubic centimetres = .02 per cent. of rosaniline acetate. A drawing of the curve is subjoined.



Our next series of experiments was carried out in a manner in all essential respects similar to the above, excepting that we now employed in every case a gramme of wool and a constant quantity (.0005 grm., *i.e.*, excess) of rosaniline salt, and reduced the time to a quarter of an hour. The remaining salt in solution was determined colorimetrically. On examining the numerical results, we found that, owing to the appearance of two subsidiary inflexed curves between about  $39^{\circ}\text{C}$ . and  $67^{\circ}\text{C}$ . (the former temperature being that of greatest observed deposition), it was not possible, without an enormous series of experiments, for which we had not sufficient leisure, to work out a complete application of Hood's law to this case. We have therefore contented ourselves with examining approximately the most interesting points, as regards dyeing effect, in this curve. We find (1) that at no practically attainable temperature near to  $0^{\circ}\text{C}$ . does colour cease to be deposited; (2) at about  $39^{\circ}\text{C}$ . the greatest amount (.09 per cent.) is taken up; and (3) at about  $82^{\circ}\text{C}$ . the curve falls lowest to the axis of no colour. Thus the results of using an excess of colour have been to greatly widen the range of temperature within which colour is taken up, to increase the general dyeing effect and to shift the point of greatest deposition (as might have been expected) about  $8^{\circ}\text{C}$ . upwards, and to doubly reverse it thereafter. The greatest influence of dissociation is, however, felt at much the same temperature as before. We have also made a short series of experiments, exactly resembling these last, with mauveine in excess. By calculating from the data nearest to  $0^{\circ}\text{C}$ ., we find that colour would cease to be taken up at  $-23.8^{\circ}\text{C}$ ., a practically unattainable temperature. At about  $49^{\circ}\text{C}$ . there is greatest deposition of colour (.08 per cent.); then there ensues a single inflexion in the curve, which want of leisure has prevented our working out in detail; and lastly, the curve descends rapidly towards the axis of no colour, though at  $85^{\circ}\text{C}$ . it is still remote therefrom.

Our experiments have convinced us that, where an aniline colour is susceptible of dissociation, there is a positive disadvantage in using high temperatures in the dye-bath. In such cases, also, the colour should, if possible, be employed only to saturation and not in excess, in order to avoid the irregularities introduced by the latter method immediately after the point of greatest deposition.

## ON THE PRODUCTION OF AMMONIA FROM THE NITROGEN OF MINERALS.

BY GEORGE BEILBY.

THE organic origin of carbonaceous deposits of all kinds from peat at one extreme to bituminous shales at the other is generally recognised, and it is also recognised that both animal and vegetable remains have contributed towards their formation. Whatever changes have occurred in the structure and composition of these remains, involving probably the loss of a great part of their oxygen, hydrogen, and carbon, a large proportion of the nitrogen of the original structures is always found in the altered deposits. It is probable that a study of the alterations which have taken place in the constitution of these nitrogenous bodies, as they occur in the older deposits, would throw further light on the whole subject, investigators having hitherto concerned themselves chiefly with the alteration of cellulose and similar substances.

In "Watt's Dictionary" (I. 426) there is a table of analyses of certain natural bitumens, which are found to contain from 1 to 2.3 per cent. of nitrogen, thus indicating their probable organic origin. In this connection it is interesting to note that while all crude oils or naphthas, artificially prepared by the destructive distillation of carbonaceous deposits, contain nitrogen in the form of alkaloidal tars, the natural American petroleum do not contain any nitrogen. This statement is based on the results of the examination of many ship loads of crude petroleum residuum which have passed through the refinery at Oakbank Works during the last twelve years. If, therefore, these petroleum have been produced from organic deposits by a process of subterranean distillation, what has become of the organic nitrogen?

It is sufficient for the object of the present paper to point out that the more or less altered nitrogenous constituents of the older organic remains behave on dry distillation in much the same way as the original substances would have done. Animal substances such as albumen, fibrine, gelatine, and the corresponding vegetable substances, yield on distillation (1) ammonia, (2) an oil rich in alkaloidal bodies, and (3) a carbonaceous residue which contains a large proportion of the nitrogen of the original substance. When the more or less altered deposits, peat, brown coal, coal, and shale are distilled, a very similar redistribution of the nitrogen is found. Ammonia is the universally-recognised product of such distillation, but the alkaloidal tars, although the repository of a considerable portion of the original nitrogen, have not received in this respect much attention. Grouven states that peat tars contain 30 to 40 per cent. of the nitrogen of the peat, and by actual determinations I have found that coal tars contain 5 to 10 per cent., and shale oils 20 to 30 per cent. A number of the more volatile alkaloids have been examined and are well known, but when all of these have been separated, others still remain a mass of uninvestigated tar.

In the years 1871-3 I investigated the distribution of the nitrogen of bituminous shales when it is distilled for the production of paraffin oils, and found that of 100 parts of nitrogen in the original shale there is found—

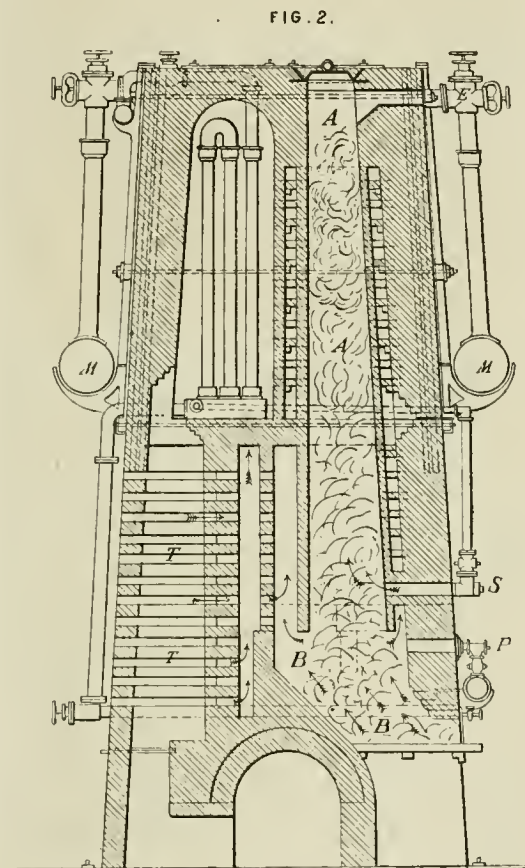
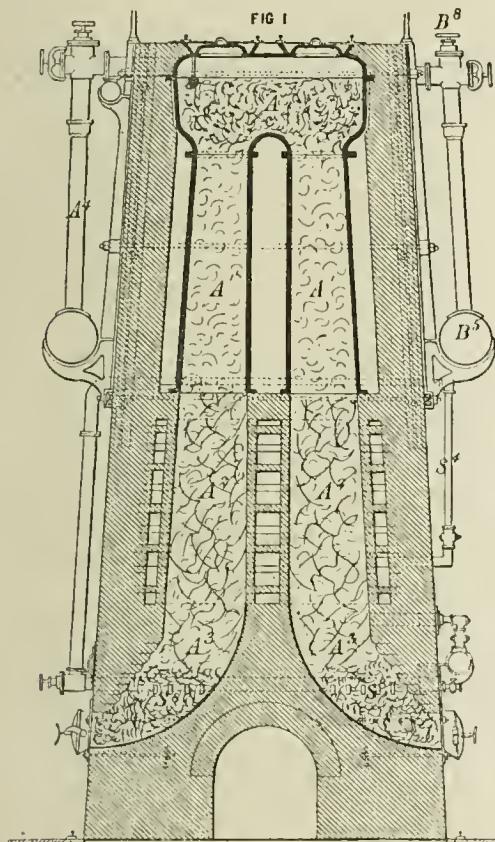
As ammonia in watery distillate.....	17.0
In the oil as alkaloidal tars.....	20.4
In the residue or coke.....	62.6

The alkaloidal tars make up about one-fourth part by weight of the oily distillate. In the products of the particular shale investigated, the oil contained 1.16 per cent. of nitrogen, while the tar contained 4

per cent. A redistillation of the entire oily distillate effects a further alteration of the distribution of the nitrogen, giving ammonia in small quantity, an oil containing less nitrogen, and a coke containing nitrogen. The coke from the crude oil stills at Oakbank Works was analysed, and it consisted of two distinct forms: (a) 1in. to 2in. of hard dense coke, containing 2·8 per cent. N; (b) 10in. to 12in. of spongy pitch, containing 3·2 per cent. N. To determine approximately the redistribution of nitrogen, which takes place when the alkaloidal tars themselves are distilled, a quantity of tar which had been separated from crude shale oil by treatment with 8 per cent. of unconcentrated sulphuric acid, was neutralised with soda and distilled. Nine fractions were taken off. The first, containing

nitrogen in the residue from the distillation of the alkaloidal tars in the works in a 5-ton still: it contained 4·1 per cent. of nitrogen.

In the distillation of shale the products come off in regular order as to boiling point and purity: First, hydrocarbon gas; next, colourless oil; then a yellow oil containing solid paraffins; and finally brown tarry oils, which consist largely of alkaloidal substances. Now, just as in the distillation of the alkaloids of the



Scale  $\frac{1}{8}$  inch to 1 foot.

water and the most volatile alkaloids, was unfortunately lost, but the remaining eight, together with the residue, were weighed and analysed by Grouven's method.

Fraction	Grammes	Contained per cent. N.
2	100	3·54
3	100	3·37
1	60	3·35
5	110	3·24
6	60	3·47
7	80	3·54
8	50	3·35
9	50	3·54
Residue	390	4·00

Before the final fraction came off the still was at a temperature little short of redness. This experiment clearly shows the comparative stability of these nitrogenous substances, the least volatile portion containing the largest proportion of nitrogen, even after exposure to a red heat. This result was confirmed, as far as the residue was concerned, by a determination of the

oil, less and less volatile matter comes off, until a residue non-volatile even at a red heat is left behind, so I believe that when shale or coal is distilled only the more volatile alkaloids come off, and the coke or residue still contains the less volatile or pitchy nitrogenous bodies. The accompanying diagram will further explain this.

DISTILLATE.	RESIDUE.
Alkaloids: $C_6H_5N$ to $C_{11}H_7N$ (?)	Higher alkaloids, or substances like carbazol, etc.
Hydrocarbons: $C_2H_4$ } to { $C_{27}H_{56}$ $C_2H_6$ } { $C_{27}H_{54}$ (?)	

It seems probable that there are series of carbon-hydrogen-nitrogen compounds of unbroken continuity from the volatile piccoline, pyridine, etc., up to



pitchy or coke-like substances. Though this pitch probably contains a considerable percentage of free carbon, yet its high percentage of nitrogen (4·1 per cent.) indicates that it must be largely composed of nitrogen-carbon-hydrogen compounds. Carbazol ( $C_{12}H_9N$ ) contains 8·38 per cent., and phenyl-naphthyl-carbazol ( $C_{16}H_{11}N$ ) contains 6·45 per cent. Both of these have been obtained from anthracene-residues, and are eminently stable substances of high melting and boiling points. It seems likely that an examination of coke, pitch, and alkaloidal residues would lead to the discovery of these or similar substances among their constituents. As time permits, I hope to extend my investigations in this direction. Mr. J. O. Morrison, in carrying out test distillations on a very small scale in the laboratory—20 to 30 grms. being heated very gradually to a good red heat—has found that the quantity of ammonia obtained simply by the action of heat in this way is double what could have been obtained on a manufacturing scale by simple dry distillation. The particular shale already referred to, distilled in the way described, gives the following results:—

	Per cent.
Total nitrogen.....	0·70
	Per cent.
Of which there was obtained as $NH_3$ ....	0·23
Of which there was obtained as alkaloids in distillate .....	0·14
Of which there was obtained in the coke 0·32	
	— 0·69
Loss .....	0·01

Taking the total nitrogen of the shale as 100 we have—

As ammonia in watery distillate .....	32·8
As alkaloids in oil .....	20·0
In the residue .....	43·7
	— 98·5
Loss.....	1·5
	— 100·0

In this case it is probable that the gradual but very thorough heating of the coke has brought about a decomposition of the residual alkaloids with a maximum production of ammonia and a minimum of free nitrogen, the large percentage of porous ash—about 90 per cent. of the whole coke favouring this decomposition. It is evident that the same heat applied to large pieces of shale might have an altogether different effect, as the gas from the interior would have to pass out through a considerable thickness of hot earthy residue, and be to a great extent decomposed. This theory would explain the well-marked difference in the behaviour of the nitrogen of pitchy minerals, such as cannel and shales. I have found that the residual cokes of the former stand a very high temperature, even a light-red heat, without dissipation of their nitrogen, while the latter are extremely sensitive to high heats, and would part with large quantities of free nitrogen at such temperatures. The investigations of Dr. H. Grouven on the conversion of combined nitrogen into ammonia have been very completely described by him (*Versuchstationen* J. 28, 343). By his method the substance is burnt in a large excess of steam, the alkaloidal vapours being conducted over or through a highly-porous “contact substance,” in which the necessary conditions of time and contact are supplied, so that they are decomposed into  $CO$ ,  $CO_2$ , and  $NH_3$ . His analytical apparatus has been in use in the laboratory at Oakbank for four or five months, and a large number of the nitrogen determinations recorded in this paper were performed with it. Three

determinations of the nitrogen in a shale by the two methods gave the following results:—

Soda-lime. Per cent.	Grouven. Per cent.
0·70 .....	0·70
0·75 .....	0·70
0·69 .....	0·63

The determination of nitrogen in a coal gave the following results:—

Soda-lime. Per cent.	Grouven. Per cent.
2·25 .....	2·20
2·20 .....	2·30

In both Grouven’s and the soda-lime process provision is made for the separate treatment of the more or less volatile portions of the substances to be analysed. In the former (Grouven’s) the alkaloidal vapours are decomposed directly by steam, the chief function of the contact substance being to keep the vapours and steam in contact at a suitable temperature; in the latter process the red-hot soda-lime in the fore part of the tube supplies the elements of water, and to a certain extent provides for contact. In the first process the non-volatile nitrogenous substances are burnt by the direct action of steam alone; in the second the elements of water are supplied by the caustic soda. But as the soda will not part with these elements unless the carbon is in intimate contact with it, this has to be insured by very careful grinding of the substance, and very intimate mixture of it with the soda-lime. Operators who have been at pains to determine the nitrogen in coals and shales must have noticed how very frequently the ignited soda-lime, on subsequent examination, shows traces of unburnt carbon, and just so surely as this is the case will the results obtained be low. When once the art of managing the heats in Grouven’s apparatus has been mastered this difficulty is completely overcome; the substance, without preliminary grinding or treatment of any kind, can be completely burnt to ash. Also, by proper regulation of the heat on the contact substance, the escape of undecomposed alkaloids can be entirely avoided.

An examination of the tables of coal analyses published in standard works has convinced me that these determinations must in many instances have been very imperfectly performed, as the averages brought out are much below the truth, as far as I have been able to ascertain it, by the careful determination of the nitrogen in specimens from many different localities. It has occurred to me that a valuable modification of the soda-lime process would be the introduction into the back end of the tube of some substance, which at a moderate heat would give off water which would not only sweep out the tube, as the carbonic acid from bicarbonate of soda at present does, but in addition would supply the elements of water to the ignited mixture of soda lime and substance in case that mixture had not been intimate enough to complete the decomposition. Hydrated oxide or sulphate of calcium would serve the purpose admirably. Any treatment of the subject of this paper would be incomplete without something more than a passing reference to Mr. R. Tervet’s researches (this journal, 2, 445) on the influence of hydrogen in favouring the production of ammonia from nitrogenous substances. These researches form a most important and original contribution to the knowledge of the subject. Having in July, 1883, been favoured with an early account of Mr. Tervet’s experiments, I arranged, with his consent, to carry out an independent series of experiments bearing on the subject. The results of these experiments were chiefly confirmatory of Mr. Tervet’s own conclusions, but had also a bearing on the line of investigation followed by Grouven, Young, and myself. They are therefore reproduced

here. The substances experimented with were coal, shale-coke, and peat. The apparatus used was an iron tube of  $\frac{1}{2}$  in. bore, heated in a gas combustion furnace. The gases passed through the tube were supplied from a graduated gasholder, and were thoroughly dried by passing through a complete set of chloride of calcium tubes. The ammonia was caught in the standard acid, in ordinary nitrogen bulbs. The coal used was the soft or partially-caking portion of the Lanarkshire main coal. Analysed both by the soda-lime and Grouven's methods it gave 2.3 per cent. of nitrogen. Mr. Tervet has expressed the ammonia obtained as pounds of sulphate per ton of coal. Stated in this way the coal used contained nitrogen equal to 243lb. of sulphate of ammonia per ton.

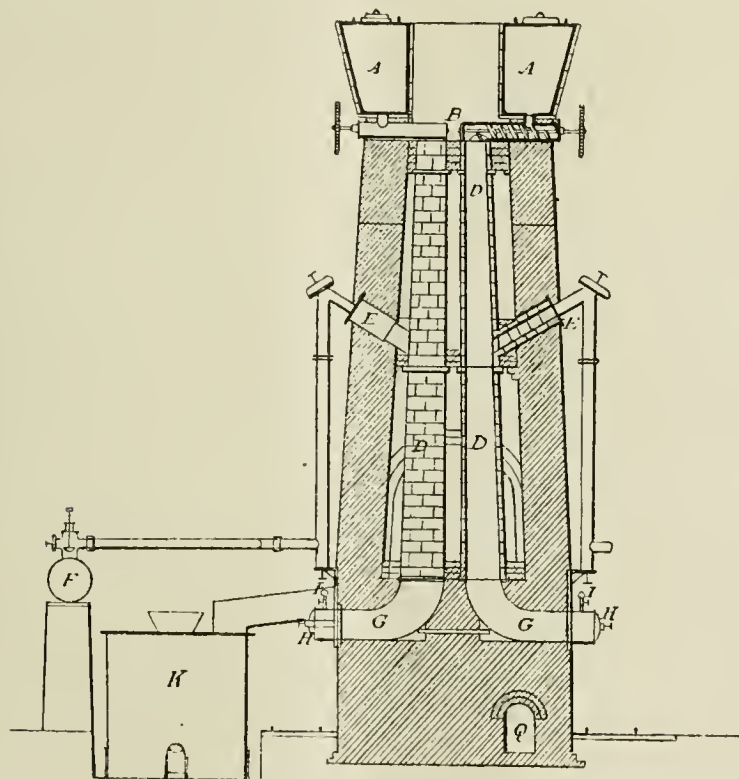
ABSTRACT OF EXPERIMENT 1.		Lb. per ton.
Distillation for 25m. ammonia as sulphate.....		27.70
1 cubic foot of $\text{CO}_2$ for 60m. ammonia as sulphate..		3.15
1 cubic foot of $\text{CO}_2$ for 55m. ammonia as sulphate..		nil
1 cubic foot of $\text{H}_2$ for 10m. ammonia as sulphate....		13.66
1 cubic foot of $\text{H}_2$ for 40m. ammonia as sulphate....		3.09
1 cubic foot of $\text{H}_2$ for 60m. ammonia as sulphate....		8.06
1 cubic foot of $\text{H}_2$ for 60m. ammonia as sulphate....		10.43
		66.39

ABSTRACT OF EXPERIMENT 2.  
Two cartridges used, measuring unitedly 14in.

		Lb. per ton.
Distillation for 25m. ammonia as sulphate.....		28.38
1 cubic foot of $\text{H}_2$ for 120m. ammonia as sulphate....		16.21
1 cubic foot of $\text{H}_2$ for 110m. ammonia as sulphate....		8.50
1 cubic foot of $\text{H}_2$ for 310m. ammonia as sulphate....		16.80
		69.92

In experiments 3, 4, and 5, peat and shale cokes

FIG. 3.



Scale  $\frac{1}{8}$  inch to 1 foot.

The coal was roughly powdered and made up in paper cartridges, which could be slipped easily into the iron tube. Each cartridge contained 250 grains (10.6grms.), and measured about 7in. in length. The cartridge having been slipped into the tube, the burners were lighted at the front end of the tube, and the heat was then carried back along the tube, just so quickly as to keep up a brisk evolution of gas. When the evolution of gas began to fall off rapidly, a small current of gas from the holder was turned on for one or two minutes, to sweep out the vapours. The nitrogen bulb was then replaced by a fresh one, and the regular stream of gas turned on, the bulb being replaced by a fresh one, whenever it was necessary to determine the ammonia.

were used, but as they were not carried beyond the stage of the two preceding experiments they need not be reproduced here. It is sufficient to note that shale coke parts with comparatively little of its nitrogen under the influence of hydrogen.

ABSTRACT OF EXPERIMENT 6.  
One Cartridge, 7in. long.

		Lb. per ton.
Distillation for 40m. ammonia as sulphate.....		29.71
Under diminished pressure, 50mm. mercury for 120m.		2.63
1 cubic foot of $\text{H}_2$ for 60m. ....		8.96
		11.35

\* The coke at this stage weighed 147.5 grains, equal to 59 per cent. of coal. It was again weighed after the steam had been passed over it, and was found practically unaltered, showing that the temperature used had been below the incinerating point.



In none of the preceding experiments had the ammonia-yielding property of the coke been perfectly exhausted, hydrogen which had passed over it still turned turmeric brown. The seventh experiment was carried out to exhaustion, and the nitrogen of the residual coke determined by soda-lime analysis. At the stage marked \* the coke was finely powdered, to insure free access of the hydrogen. Throughout all of these experiments the heat of the combustion tube was very uniform: it showed as a well-marked blood red in diffused daylight.

## ABSTRACT OF EXPERIMENT 7.

One cartridge, 7in. long, 1in. diameter.		Total
Distillation for	Per ton.	N=100
3 cub. ft. of H <sub>2</sub> for 20m.	27.00lb.	= 11.11
1 cub. ft. of H <sub>2</sub> for 20m.	26.65lb.	
1½ cub. ft. of H <sub>2</sub> for 20m.	4.66lb.	
1½ cub. ft. of H <sub>2</sub> for 18m.	5.73lb.	
1½ cub. ft. of H <sub>2</sub> for 24m.	2.81lb.	
Ammonia as sulphate		= 16.11
Total by distillation and hydrogen		66.88lb. = 27.52
N in tar distillate (1.5% on 5% of tar)		7.84lb. = 3.22
Left in coke by soda-lime determination (0.82%)		50.62lb. = 20.83
		125.31lb. = 51.57
Lost as free N		117.66lb. = 48.43
Total N of original coal=ammonia as sulphate		213.00lb. = 100.00

The coke (containing 0.82 per cent. N) was heated to whiteness in the laboratory in an iron crucible, and was afterwards analysed by soda lime, and gave N=0.4 per cent. It was again heated in a fire-clay crucible in the smith's fire, and kept at a bright white-heat for several hours. Analysed by soda lime it gave N=0.25 per cent. Treated with hydrogen at a red heat at this stage it gave up no ammonia. These latter results indicate the wonderful stability of some of the nitrogenous constituents of the coke. As Mr. Tervet is still following out his investigations on this subject, I refrain from further comment on these experiments. I have devoted much time and attention during the last twelve years to the solution of the problem, how more economically to dispose of the nitrogen of shales and coals. Both alone and also in association with Mr. William Young many experimental investigations have been carried out, some of the practical results of which have been embodied in manufacturing processes now commercially established. Certain of these have been described elsewhere (*Journal of Gas Lighting* 40, 257, and 42, 317, *Trans. M. Inst. S.* 5, 158), and need, therefore, be only very generally noticed here. For many years steam has been passed through shale retorts during the distillation, and has been found to increase the yield of ammonia to a considerable amount beyond what had been obtained by simple dry distillation. In 1873, at Magdalen Bridge Works, Mr. Young and the writer proved that the same effect was produced if steam was passed through the red-hot residue after the oil distillation was completed. This system was afterwards applied to a bench of Young's patent retorts in Oakbank Works. But though steam was used in these ways with distinct advantages to the yield of ammonia, the distribution of the nitrogen in the products was still unsatisfactory. The results of this treatment with the shale, already referred to, gave the following distribution, again taking the total nitrogen of the shale as 100—

As ammonia in the watery distillate	21.2
In the oil as alkaloidal tars	20.1
In the residue or coke	58.3

As the nitrogen of the coke could be obtained as ammonia by ignition of the coke with soda lime, it occurred to us that if the coke were ignited in steam

the same effect should be produced. This was proved to be the case by actual experiment. In 1881 a single retort was erected at Oakbank, in which shale was first distilled at a low-red heat in an iron retort, and was afterwards subjected to the action of steam at a bright-red heat in a fire-clay retort. The fixed carbon of the residue was burned away, and the nitrogen came off as ammonia. In this retort almost theoretical results were obtained, the exhausted residue or ash containing only 4.9 per cent. of the nitrogen of the shale, while the remainder was found in the distillate. Stating these results as before, we have—

As ammonia in the watery distillate	74.3
In the oil as alkaloidal tars	20.1
In the residue or coke	4.9

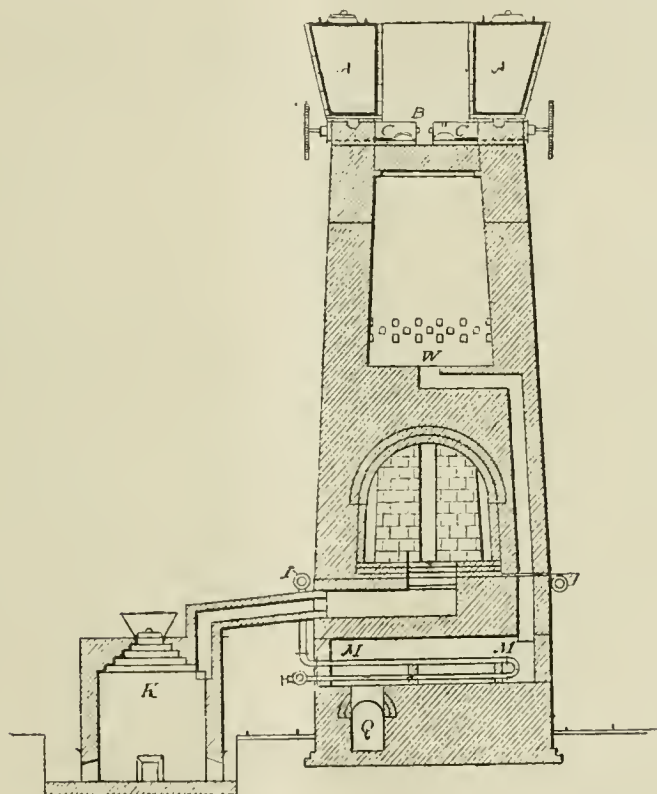
With the exercise of appropriate care it was found possible to repeat these results at will, but a serious difficulty was experienced in working certain shales, owing to the fusibility of the ash after the removal from it of the fixed carbon. Our earlier retorts were designed of small diameter and capacity, to insure the ready access of heat to the centre of the mass of shale, and were worked at a temperature little, if at all, short of the melting or softening point of the ash. We have more recently found it better to make the retorts of much larger size, so that the shale may remain under the influence of heat and steam for a longer time, and at a somewhat lower temperature. In this way much of the difficulty which arises from the melting and sticking of the ash is avoided. The shale retorts shown in cross section in Fig. 1 were erected at Pentland Works a year and a half ago, and have given very excellent results during that time. The shale is charged into the hopper A by doors on the top. It is here heated up by the hot gases passing up from the retorts below, and as the whole column of material is moved down by the removal of a part of the spent ash at the bottom, the shale passes in succession through the iron retorts A<sup>1</sup> and the fire-brick ovens A<sup>2</sup>. In the former it parts with its hydrocarbon oils and paraffin, and in the latter the carbon is more or less completely burned away in steam, producing water gas and ammonia. The earlier forms of these retorts were heated by open furnaces, and the proper regulation of temperature was very difficult. In the retorts at Pentland the furnaces are replaced by coal retorts, shown in cross section in Fig. 2. In these combined retorts and gas producers coal is first distilled, then partly burned in steam, and finally completely reduced to ash by the regulated admission of air. At each stage heating gas is produced, and by suitable appliances is burned in the chambers in which the retorts are set. By this arrangement the full advantages of gas firing are gained, and at the same time a large yield of by-products is obtained from the fuel.

In 1882 we discovered that a certain proportion of air might be mixed with the steam used for the burning of nitrogenous carbon, without seriously reducing the yield of ammonia. The principal advantage of such a use of air is that a certain amount of heat is thereby generated within the retort, and consequently less has to be supplied from the outside, and a lower working heat can therefore be maintained. This use of air, if properly regulated, does not sacrifice much of the nitrogen of the coke, and we have found in certain cases that the increased volume of hot gas from the lower retort, in which the carbon is being consumed, tends to carry on the distillation of the shale in the upper retort, under conditions very favourable to the production of the most valuable hydrocarbons—the solid paraffins. In January, 1883, a set of retorts, worked alternately with steam alone and with steam and air, showed with the latter a gain of oil yield of about 10 per cent.,

and of solid paraffins of 25 per cent.—that is, a shale which with steam alone gave 30 gallons per ton of an oil containing  $10\frac{1}{2}$  per cent. of solid paraffins, with steam and air gave 33 gallons per ton of an oil containing  $12\frac{1}{2}$  per cent. of solid paraffins. The steam used in both cases gave about 72 gallons of condensed water per ton of shale. In other sets of retorts a similar gain of oil and paraffins has been obtained by the use of a sufficiently large volume of steam alone, 120 to 150 gallons of water being condensed per ton. It is at present an open question which of these methods will in the long run give the most economical results. The advantages of air are, its cheapness as compared with steam, the internal heat which it supplies, and a certain crispness which it gives to the spent shale, which permits

long we shall recover the nitrogen of the alkaloidal tars by the use of the beautifully-contrived ammonium ovens of Dr. Grouven. A description of his apparatus, comprising incinerating and ammonium ovens and ammonia soda plant, would alone furnish matter for a long paper, and cannot be entered into on this occasion. The interest of a process confined to a comparatively limited industry like the oil trade of Scotland cannot for a moment be compared with that attaching to its application to the larger industry of fuel gas making. A good deal of our attention has been turned on the application of the Grouven-Young-Beilby process to the treatment of coal for the production of heating gas and ammonia. When it is remembered that the process involves the decomposition of water by carbon, the oxidation of

FIG 4



Scale  $\frac{1}{2}$  inch to 1 foot.

of its being withdrawn from the retorts very easily. The disadvantage of the use of air is the increased volume of incondensable gas which has to be handled, largely consisting, as it does, of the useless nitrogen of the air. Not merely does this extra volume involve increased exhausting and condensing plant, but it carries forward with it an excessive quantity of light naphtha which can only be recovered by the use of very extensive oil scrubbers. Only a prolonged working experience will enable us to decide as to the economical limits of the use of air in shale distillation. The thermo-chemical and economic aspect of its use in the treatment of coal will be referred to later.

The process so far described has only had reference to the recovery of the nitrogen of the residual coke from shale distillation, but it is probable that before

the latter to carbonic oxide and carbonic acid, and the setting free of hydrogen, its suitability as a means of producing heating gas is evident, as far, at least, as the quality of the gas is concerned. When it is further pointed out that by this process from 60 to 70 per cent. of the nitrogen of the coal can be obtained as ammonia, it must be admitted that it opens up a distinct possibility of an economical production of heating gas. Between the theoretically perfect process—so well illustrated by the analytical apparatus of Grouven—and its economical realisation on a large scale there are many steps to be taken. The fundamental difficulty of the transition is simply stated. It is this: The temperature at which carbon freely decomposes water is considerably above that at which ammonia is decomposed. The former tempera-



ture may be taken as not lower than  $1,100^{\circ}$  to  $1,200^{\circ}$  C. The recently-published results of Drs. Ramsay and Young (*Chem. Soc. Journ.* 256—88.) show that under favourable circumstances (contact surface and time) decomposition begins a little below  $500^{\circ}$ . It is evident, therefore, that favourable circumstances and a temperature of  $1,100^{\circ}$  to  $1,200^{\circ}$  will completely decompose ammonia. Clearly, then, the ammonia could only be saved by making the conditions of the process as unfavourable as possible to its decomposition. It was necessary therefore to reduce the chances of contact of the ammonia molecules with surfaces at the decomposing temperature. This could best be done by diluting the ammonia gas with a large volume of a neutral gas consequently Grouven provides in his analytical to apparatus the use of a quantity of steam equal to 20 to 30 times the weight of the substance to be analysed. It is evident that such a proportion could never be economically used on a large scale. Anything like 20 tons of steam for 1 ton of coal would place the process outside of the practical region at once. In our earlier laboratory experiments we found that about 60 per cent. of the total nitrogen could be obtained as ammonia by the use of 4 to 5 parts of steam

tar vapours are completely decomposed. Only a pitchy dust is deposited in the long dust pipes and mains. The dross used in these retorts contains nitrogen equal to 165lb. to 170lb. of sulphate of ammonia per ton. Week after week since the beginning of the year we have obtained yields ranging from 90lb. to 125lb. of sulphate per ton, or say 60 to 70 per cent. of the total nitrogen. The quantity of steam used has varied from 2,500lb. to 3,500lb. per ton of dross, equal to 250 to 350 gallons, so that the proportions of coal to steam now stand—1 coal to  $1\frac{1}{4}$  steam. The composition of the incondensable gas varies somewhat, according to heats, air supply, etc. A sample collected on the 5th inst., and analysed, showed the following composition (by volume):—

CO <sub>2</sub> .....	16.6
CO .....	8.1
CH <sub>4</sub> .....	2.3
H <sub>2</sub> .....	28.6
N <sub>2</sub> .....	44.4
	100.0

This indicates a rather excessive air supply, as we aim generally at a consumption of one half of the fixed carbon with air, the other half with steam.

TABLE I.

*Showing the Products of 1 ton of Coal when 50 per cent. of its Carbon is incinerated with Steam and 50 per cent. with Air.*

Composition of Coal.			Steam.	Air.	Chemical Reactions.	Products.		Vol. of Gases at 500°.		Vol. of Gases at 16° C.	
	per cent.	lb.	lb.	lb.			lb.	cub. ft.	per cent.	cub. ft.	per cent.
Carbon	—	879	—	—	3C+5H <sub>2</sub> O	Carbonic Oxide	2731.6	104.130	15.98	36.795	21.32
	78.53	879	2197.5	—	=CO+2CO <sub>2</sub> +5H <sub>2</sub>	Carbonic Acid	2118.7	52.355	8.03	18.500	10.72
	—	—	—	5096	2C+O <sub>2</sub> =2CO						
Hydrogen	5.61	126	—	—		Hydrogen	338.6	179.584	27.57	64.164	37.19
Nitrogen	1.00	22	—	—		Nitrogen	392.1	150.327	23.07	53.082	30.77
Ammonia						Ammonia	17	1.075	.16	—	—
Sulphur	1.11	25	—	—		Sulphuretted Hydrogen	26.6	.826	.13	—	—
Oxygen	9.69	217	—	—		Water	244 2500	163.075	25.03	—	—
Ash	1.03	92	excess 2500	—							
	100.00	2240+	4697.5+	5096		Ash	92	—	—	—	—
							120 3.5	651.372	100.00	172.541	100.00

to 1 of coal. Then it occurred to us that air might be made to take the place of a part of the steam, and experiments proved the correctness of the surmise. Thereafter our aim was to devise a practical apparatus in which the conditions most favourable to the preservation of the ammonia could be economically maintained. Figs. 3 and 4 show two cross sections of a bench of coal retorts, which have been at work at Oakbank since October last. In the upper part of the retort the dross or small coal, which is fed into the top by screw feeders, is distilled, the tar vapours, together with the steam, passing down through red-hot coke, are decomposed into permanent gas and ammonia, and pass away by the exit pipe situated about the middle of the retort. The coke passing down into the lower half of the retort is burned in a mixture of steam and air, the resulting gases passing upwards and away by the centre exit pipe. The united gases from the upper and under retorts are drawn through condensers and scrubbers, where the ammonia is separated and recovered. When the heats are properly regulated the

With such an admission of air the composition of the gas would probably be:—

CO <sub>2</sub> .....	21.32
CO .....	10.72
CH <sub>4</sub> (etc.) .....	nil
H <sub>2</sub> .....	37.19
N .....	30.77
	100.00

Elsewhere (*Trans. M. I. Scotland*, 5, 178) I have published a series of tables showing the thermal values of the reactions between the elements of an average coal and steam in excess. The accompanying Tables, I., II., and III., show these reactions and values when 50 per cent. of the carbon is burned with air and 50 per cent. with steam. The units used are Centigrade degree-pounds. The thermal values of the equation are based on Thomson's numbers:—

[C—O]=96,960.
[C—O]=29,290.
[CO—O]=67,670.
[H <sub>2</sub> —O]=53,069, at 100°.

The available published figures for the loss of heat through the brickwork of retort benches are so mani-

festly inaccurate that new determinations of the conducting power of fire-brick were made. The method employed was similar to that used by Peclet (*Ann. Ch. Phys* 3, ii., 107), but instead of using the small differences of temperature to which one is restricted when water is used on both sides of the conducting medium, large differences, of  $260^{\circ}$  to  $300^{\circ}$  C., were maintained by the use of a lead-tin heating bath. A detailed description of these experiments would be out of place here, but the general result showed that one square foot of fire-brick 1 in. thick passed 6.59 heat units per hour for each degree of difference between the temperatures of the two sides of the diaphragm. From this it was calculated that the loss over the whole apparatus amounted to about 2,000,000 units per ton of coal treated. From an examination of Table II. it is seen that 6,961,362

TABLE II.

*Showing the Heat Units Absorbed in the Partial Incineration of Coal with Excess of Steam.*

To heat 2,240lb. of coal to $1,000^{\circ}$ C. (a) .....	$2240 \times 24 \times 1000$	537,600
To raise 4,697lb. of water into steam at $100^{\circ}$ C. (b) .....	$4697 \times 5 \times 620$	2,912,470
To raise 4,697lb. of steam from $100^{\circ}$ C. to $1,000^{\circ}$ C. (c) .....	$4697 \times 5 \times 47 \times 900$	1,986,831
Heat absorbed in the reaction $3C + 5H_2O = CO + 2CO_2 + 5H_2$ (d) ....	—	1,648,125
To maintain the temperature of the apparatus against losses by conduction and radiation (e) .....	—	2,000,000
		9,085,026
Deduct heat of reaction C, $O^2$ (f) ....	$879 \times 2416$	2,123,664
Total heat units to be supplied ..	—	6,961,362

TABLE III.

*Showing the Comparative Heating Value of the Original Coal and of the Gases produced by the Incineration of 50 per cent. of its Carbon with Excess of Steam, and 50 per cent. with Air, deducting from the latter the Heat absorbed in their production.*

Pounds.		Heat Units per lb.	Heat Units	Heat Units
2240	Coal.....	7000	—	15,680,000
2734.6	Carbonic Oxide	2427	6,563,040	—
383.6	Hydrogen.....	29,035	11,512,400	—
			18,075,440	—
	Deduct heat absorbed as shown in Table II. ....		6,961,362	11,114,078
	Loss in heat value of 1 ton of coal by gasification, equal to 29.11 per cent. ....		—	4,563,922

units have to be supplied for the gasification of one ton of coal. Taking up the several items *a*, *b*, *c*, *d*, *e*, and *f*, it is found that (*a*) is provided by waste heat from the later stages, (*b*) is expended in boilers or steam generators or supplied by waste heat, (*c*) and (*d*), minus quantities, are met by (*f*), leaving only 1,511,292 units to be supplied otherwise, (*e*) has to be provided for in the apparatus, (*c* + *d* + *e* - *f*) may be provided in either of two ways, or by a combination of the two—first, by pre-heating the steam and air, or secondly by passing in heat through the walls of the retorts. The first has been adopted by Grouven in his most recent incinerating ovens. The mixture of steam and air is raised to a white heat by means of hot blast ovens or stoves on the exchanging principle, and the hot blast is forced through the peat or coal in solidly-built ovens. The second plan has been adopted primarily in the retorts shown in Figs. 1, 2, 3, and 4, supplemented, however, by the first. It seems

to us, in the light of our present knowledge, that the combined system is the more economical, but an extended experience may lead to a change of opinion. As to the practical heating results obtained—the small set of retorts at Oakbank are consuming in all 4 tons of coal per day, and in return produce 90lb. to 100lb. of sulphate of ammonia per ton, and heating gas sufficient to fire 32 shale retorts, which were formerly heated by two ordinary furnaces, consuming each 2 tons of coal per day. While these data are not sufficiently full to enable one to frame a *final* estimate of the practical gains and losses of heat, they do give a very clear indication that practical results are being realised which correspond very fairly with theoretical calculations. For the sake of simplicity this apparatus was not provided with any adequate heat recuperators, by the use of which, in future, we may expect to effect great economy of heat.

In concluding this paper it gives me much pleasure to acknowledge my indebtedness to my friends and colleagues, Messrs. G. D. H. Mitchell, E. H. Ronalds, and J. O. Morrison, who have performed numerous analyses and laboratory experiments in connection with the subjects treated of, and to Messrs. W. A. Bryson and G. J. Hamlen, who have superintended and carried out experimental operations in the works, at no small sacrifice of their personal leisure and comfort.

## DISCUSSION.

The CHAIRMAN: I am sure we are all deeply indebted to Mr. Beilby for this very remarkable paper, for he must have spent a great amount of labour upon it. The apparatus and diagrams which we see before us are extremely ingenious, and realise all that has been predicted for the process. There must be several members present who wish to say something on this very important paper, and we shall be glad to hear their remarks.

Mr. R. R. TATLOCK: I do not know that I can say anything that will add to the interest of this very valuable paper, and what little I have got to say is entirely from an economical point of view. I must confess that when I first heard of Mr. Beilby's improvements it occurred to me that there was no need for incurring expense for steam and outside heating in order to obtain ammonia, there being so much of it wasted in this country by iron manufacturers and others who had it absolutely for nothing. In the blast furnaces of Scotland, working with raw coals in Scotland, and in the comparatively few working with raw coal in England, we have a waste of ammonia amounting to 30,000 tons per annum. That ammonia can be had for the extracting, for the mere condensing or fixing by means of chemicals, which can be done, even at present low prices, with a manufacturer's profit of at least 100 per cent. These processes have not been carried out to any great extent, even up to the present time. In addition to that, there is a large quantity of ammonia, as we all know, wasted in the present processes of coking coal, that up to the present moment has only been very partially recovered. We cannot say that that enormous amount can be got merely for the lifting, because it seems that in order to recover it the ovens must practically be rebuilt before the best results can be obtained, but looking to the enormous consumption of other nitrogenous materials, it is quite plain that there is plenty of room for all the processes. There were last year about half a million tons of nitrate of soda consumed over the entire world. If we take, as I have said, the waste of 30,000 tons going on at present—I cannot say how much from gas producers, and that must also be considerable—and perhaps 200,000 tons which could be recovered from coking ovens, there is still a large



margin, and the question is whether this process of Mr. Beilby's, where extra expense is involved, will pay at the present rates—which are likely to be continued for a length of time, probably for many years. Since it is not probable that nitrate of soda will be brought into European ports very much cheaper than at present, I think there is a prospect of plenty of room for the ammonia that can be produced by these means, over and above what is absolutely wasted, so that even should these wastes be utilised, there will still be plenty of outlet for ammonia manufactured in this way, and if Mr. Beilby can satisfy us that it is an economical one, with sulphate of ammonia at present prices, or even 25 per cent. lower, there is no reason why it should not be adopted with success, even considering that it does cost money to produce it, and has to contend with ammonia already produced, and going to waste, as well as with nitrate of soda.

The CHAIRMAN: I was struck with the large proportion of nitrogen that appeared to be obtained—40 per cent., I think.

Mr. BEILBY: That is on the authority of Dr. Grouven. He asserts that 30 to 40 per cent. comes away in that form. From my own examination of peat I should say that this is probably an over-statement, but in certain shales I have found fully 40 per cent. of the original nitrogen in the oily distillate.

Dr. WALLACE: The tenacity with which nitrogen is retained by carbon, as described in Mr. Beilby's paper, is well illustrated by the case of the animal charcoal used in sugar refineries. The new charcoal, containing about 10 per cent. of carbonaceous matter, usually described as carbon, has from 1·2 to 1·5 per cent. of nitrogen, equal to 12 to 15 per cent. of the weight of carbonaceous matter. Every time this charcoal is used in the refining process it is washed, dried, and reburnt, and on each occasion there is a loss of nitrogen, somewhat considerable during the first few times but afterwards very small. Even after 200 reburnings, occupying three to four years, the proportion of nitrogen which remains constitutes 2 to 3 per cent. of the weight of the carbonaceous matter. Formerly it was the custom in some sugar-houses to draw the charcoal red hot from the kilns, and extinguish it with water, when invariably ammonia was given off along with the steam, the quantity being very considerable when the charcoal was comparatively new. The subject which Mr. Beilby has brought before us is one of great importance, and, as Mr. Tatlock has said, there is no doubt about the process itself, but experience is required to show whether it is more economical than methods in use in oilworks of older construction.

Mr. TERVET: I have much pleasure in expressing the satisfaction with which I have listened to Mr. Beilby's paper, which I consider a most valuable contribution to the subject, and one in which I have taken a deep interest. In acknowledging the honour which Mr. Beilby has done me in referring to my own investigations, I have to observe that his results differ somewhat from those obtained by myself; and I believe that these differences arise from his not having passed a sufficient volume of hydrogen through the retort. Mr. Beilby said, I think, that during the first 240 minutes one cubic foot of hydrogen was passed, and during the second 240 minutes three-quarters of a cubic foot. In my own experiments, during one-half that time, over a hundred times that volume of hydrogen would be passed, and under slight pressure. In describing my experiments I made special reference to the importance of having a very large excess of hydrogen in circulation, therefore I think that the small volume of hydrogen is the cause of the discrepancies which exist between the results obtained by Mr. Beilby and my own. I have also to say that

I can fully bear out the results of Mr. Beilby's investigations on the alkaloidal bodies obtained from crude shale oils. It is a very interesting fact that the proportion of nitrogen should be distributed almost uniformly through the whole series of a fractional analysis of the distilled alkaloids.

Mr. T. L. PATERSON: I have always found that new animal charcoal contains a large proportion of nitrogen, that leaves it after the charcoal has been used, and yet after a great many years it still contains portions of nitrogen. If Mr. Beilby had made any experiments with the view to determine the nature of the alkaloids remaining in the coke from the distillation of shale, it might throw considerable light upon the presence of nitrogen in the charcoal.

Mr. BEILBY: I have not direct proof, the evidence is only circumstantial.

Mr. PATERSON: I think your hypothesis is a very probable one, but I just wanted to know if you had made any such experiment.

—♦♦♦♦♦♦♦♦♦♦—

*Meeting held on Thursday, March 27th, 1884.*

MR. ED. C. C. STANFORD IN THE CHAIR.

## ON THE DISTRIBUTION AND CONDENSATION OF THE GASES IN THE LEADEN CHAMBER.

BY J. MACTEAR, F.C.S.

It is somewhat surprising that so little work has been done towards the elucidation of the relative distribution and condensation of the gases in the various parts of the leaden chambers employed in the manufacture of vitriol. The most complete series of experiments, of which details have been published, are those of the late Mr. H. A. Smith, who made a considerable number of experiments upon the subject both in the laboratory and in the chambers themselves. The result of his experiments led him to believe that the chief portion of the acid was produced and condensed close to the surface of the acid already formed in the chambers. He made his experiments on a chamber 140ft. long by 30ft. high and 25ft. wide, and he divided the chamber into lengths of 10ft., and into levels or zones of 3ft. and 15ft. from the floor. I have taken out his figures and have put them into the form of diagrams, which show the relative proportions existing at the various points of the chamber of sulphurous acid, sulphuric acid, and nitric acid. One of the chief points brought out by thus placing his figures in diagrammatic form is, that we can see in a very marked degree the effect

TABLE I.

*Distance in Feet from Entrance of Gases.*

	10	20	30	40	50	60	70	80	90	100	110	120	130	140	
SO <sub>2</sub> .....	72	71	46	32	25	26	30	22	29	22	23	13	18	18	At 15 feet.
SO <sub>3</sub> .....	0	0	6	18	23	20	18	16	19	12	12	7	7	10	
N <sub>2</sub> O <sub>5</sub> ...	25	13	13	13	8	7	14	13	16	20	7	3	6	6	
Totals	97	89	65	63	56	53	62	51	64	54	42	23	31	34	
SO <sub>2</sub> .....	3	8	16	29	28	18	19	20	17	17	14	13	8	16	At 3 feet.
SO <sub>3</sub> .....	81	89	76	70	68	67	60	56	48	30	38	30	26	33	
N <sub>2</sub> O <sub>5</sub> ...	8	3	6	4	4	12	8	17	20	26	26	15	12	3	
Totals	92	100	98	103	100	97	87	93	85	73	78	58	56	52	

of the introduction of the steam, which has reduced largely the amount of sulphurous acid at the points where it enters the chamber, there being a corresponding increase in the amount of the sulphuric acid. Taking, now, Mr. Smith's results in figures, we find that there are great variations and irregularities, due, I believe,

TABLE II.

	GLOVER TOWER.							GAY-LUSSAC TOWER.	
	Gases Entering Centesimally.	1st Chamber. Gases Entering Centesimally.	2nd Chamber. Gases Entering Centesimally.	3rd Chamber. Gases Entering Centesimally.	4th Chamber. Gases Entering Centesimally.	5th Chamber. Gases Entering Centesimally.	6th Chamber. Gases Entering Centesimally.	Gases Entering Centesimally.	Escaping into Chimney Centesimally.
SO <sub>2</sub> .....	6·325	6·318	4·461	2·630	1·402	·704	·261	·035	·035
O required to convert SO <sub>2</sub> into SO <sub>3</sub> .....	3·155	3·151	2·220	1·302	·686	·336	·113	—	—
N equivalent to O fixed in Fe <sub>2</sub> O <sub>3</sub> and SO <sub>3</sub> ....	45·620	45·206	46·598	47·971	48·892	49·415	49·748	49·917	49·978
O equal to air in excess .....	9·411	9·403	9·693	9·978	10·169	10·278	10·347	16·382	10·395
N equal to air in excess .....	35·486	35·803	36·905	37·993	38·722	39·137	39·400	39·531	39·582
N <sub>2</sub> O <sub>4</sub> .....	—	·119	·123	·126	·129	·130	·131	·132	·010
Totals .....	100·000	100·000	100·000	100·000	100·000	100·000	100·000	100·000	100·000

TABLE IV.\*

Comparative Chamber Condensation of Various Series of Sulphuric Acid Chambers.

NO. OF CHAMBER.	A in sequence.	B in sequence.	C in sequence.	D in sequence.	E in sequence.	F 1 and 2 into 3.	G in sequence.	H in sequence.	I in sequence.	K in sequence.	L in sequence.	M in sequence.	N in sequence.	O 1 into 2 into 3 into 4 into 5 into 6.	P in sequence.	Q in sequence.
1.....	31·50	32·20	32·5	38·4	34·1	33·83	52·42	63·85	38·7	78·57	53·95	81·82	89·68	27·2	26·1	32·0
2.....	29·27	30·26	24·8	35·2	20·03	42·22	26·38	36·15	33·6	21·43	36·90	17·78	*5·15	15·9	31·3	29·0
3.....	18·71	24·11	19·3	15·6	19·66	22·61	16·53	—	12·9	—	9·15	—	*5·17	24·8	20·4	17·5
4.....	10·32	7·04	15·5	6·2	18·15	—	—	—	9·8	—	—	—	—	20·2	11·8	13·7
5.....	6·45	5·00	5·8	3·2	7·25	—	—	—	3·4	—	—	—	—	7·3	7·9	5·4
6.....	3·75	1·39	2·1	1·4	·54	—	—	—	1·6	—	—	—	—	4·1	2·4	2·4
7.....	—	—	—	—	·27	—	—	—	—	—	—	—	—	—	—	—
Tunnels .....	—	—	—	—	—	1·34	4·67	—	—	—	—	·40	—	—	—	—
	100	100	100	100	100	100	100	100	100	100	100	100	100	100	99·9	100

TABLE V.

Nineteen Weeks' Average—No Covers used on Trays.

Gauge Position.	No. 1 Chamber.		No. 2 Chamber.		No. 3 Chamber.		No. 4 Chamber.		No. 5 Chamber.		No. 6 Chamber.	
	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.
Roof .....	100	125	100	140	100	130	100	109	100	84	100	88
Middle .....	100·1	122	95·2	132	13·5	128	81·4	111	106	79	72·1	87
Bottom .....	102·4	123	103	136	113·8	127	87·5	115	110·9	70	63·0	81

TABLE VI.

Nineteen Weeks' Average—No Covers used on Trays.

CHAMBERS.	1	2	3	4	5	6
Per 100 of Acid made in ....						
Top Tray .....	33·0	33·0	30·5	37·1	31·5	42·3
Middle Tray .....	33·1	32·2	34·6	30·2	33·5	30·5
Bottom Tray .....	33·9	34·8	34·9	32·7	35·0	27·2
Percentage of Total Acid made in each Chamber .....	26·1	31·3	20·4	11·8	7·9	2·4

\*In \*N the chambers are irregular in size, Nos. 2 and 3 being together only about one-third the capacity of No. 1. In §O the chambers are all of the same size, and are worked in connection with two sets of kilns, the gas from one set passing into chamber No. 1, and thence into No. 2, the gas from the other set of kilns passing into chamber No. 3, thence into 4, and the residual gases from both Nos. 2 and 4 passing into No. 5, and then finally into No. 6.



TABLE VII.

*Weekly Average—Covers in use, close up to Roof of Chambers.*

Position.	No. 1 Chamber.		No. 2 Chamber.		No. 3 Chamber.		No. 4 Chamber.		No. 5 Chamber.		No. 6 Chamber.	
	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.
Roof .....	100	128	100	139	100	103	100	85	100	70	100	80
Middle .....	105.8	131	89	131	111.7	106	77.4	80	71.3	64	67.8	70
Bottom .....	83.6	128	112.5	132	112.4	104	97.3	84	75.2	70	72.1	73

TABLE VIII.

*Weekly Average—Covers in use close up to Roof of Chambers.*

CHAMBERS.		1	2	3	4	5	6
Per 100 of Acid made in ....	Top Tray .....	34.54	34.11	30.85	36.4	40.0	40.4
	Middle Tray .....	27.5	34.16	28.2	29.0	28.2	28.2
	Bottom Tray .....	28.91	38.4	31.69	35.4	31.0	31.4
Percentage of Total Acid made in each Chamber .....		38.4	35.2	15.6	6.2	3.2	1.4

TABLE IX.

*Weekly Average—Covers in use, placed one foot above Trays.*

Gauge Position.	No. 1 Chamber.		No. 2 Chamber.		No. 3 Chamber.		No. 4 Chamber.		No. 5 Chamber.		No. 6 Chamber.	
	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.	Ratio.	* Tw.
Roof .....	100	125	100	132	100	123	100	116	100	104	100	103
Middle .....	95	123	70	119	106	125	87	115	95	102	85	98
Bottom .....	84	120	99.6	128	120	126	93	115	92	98	97	100

TABLE X.

*Weekly Average—Covers in use, placed one foot above Trays.*

CHAMBERS.		1	2	3	4	5	6
Per 100 of Acid made in ....	Top Tray .....	36.18	37.7	31.7	36.3	35.6	34.6
	Middle Tray .....	33.8	26.5	33.6	31.0	32.8	32.4
	Bottom Tray .....	30.02	35.8	31.7	32.7	31.6	33.0
Percentage of Total Acid made in each Chamber .....		32.0	29.0	17.5	13.7	5.4	2.4

to the fact that various samples have been used for the estimations, and that they have not been drawn

TABLE III.

*Comparative Chamber Condensation.*

No. of Chambers.	Acid made.	H <sub>2</sub> SO <sub>4</sub> in Acid made.	Excess Water.	Per cent. of H <sub>2</sub> SO <sub>4</sub> made in the various Chambers.
1	Tons.	Tons.	Tons.	
2	23.52	19.89	3.63	32.20
3	22.59	18.68	3.91	30.26
4	20.35	14.89	5.46	21.11
5	10.23	4.35	5.88	7.04
6	5.81	3.09	2.75	5.00
	2.19	.86	1.33	1.39
Totals.	84.72	61.76	22.96	100.00

simultaneously, as would be absolutely necessary to obtain accurate results.

In order that we may have some idea of the conditions under which the chambers are worked as regards

the distribution in each chamber of the various gases, let us take the case of a set of sulphuric acid chambers, six in number, burning pyrites, and assume that the gases are led, first, through a Glover tower, and then through the chambers, one after another, in sequence, finally passing through a Gay-Lussac tower. Let us also assume that the nitrous compounds present in the chamber are = 10 per cent. of nitrate of soda on the sulphur burned; that these compounds are calculated as existing as N<sub>2</sub>O<sub>4</sub>; that the residual gases are allowed to escape with 10.4 per cent. of oxygen, 33 grains per cubic foot nitrous compounds calculated as nitrate of soda, and 5 per cent. of the original sulphur escaping as SO<sub>2</sub>. Neglecting the watery vapour, which it is very difficult to calculate, Table II. shows the relative composition of the gases entering the Glover tower, and as they pass from chamber to chamber in the series. The difference between the gases entering the first chamber and those entering the second

\* This figure is merely taken for convenience of calculation. Oxygen varies much in the different cases. A fair average might be taken as 7 to 7½ per cent. estimated in the exit gases after passing all condensing towers, etc. With old or leaky chambers it often amounts to as much as 11 per cent.

chamber show at a glance the amount of condensation which has taken place. A more striking way, however, of showing this is, perhaps, by the diagram, in which the large square represents the total chamber space and the lower shaded square represents the proportionate bulk of sulphurous acid in the various chambers as the gases pass through. The upper shaded squares show the nitrous compounds, as  $\text{NO}_2$ , in the same way. The results from which these figures were calculated were obtained by a long series of determinations of the acid condensed in the various chambers, and which were made with great care, with the special object in view of finding out the relative amounts condensed in the various chambers. Table III. shows comparative chamber condensation, and gives the results for one week, during which the loss of acid escaping was estimated by my continuous testing arrangement, and for the week in question it amounted to 1.149 per cent.  $\text{H}_2\text{SO}_4$  of sulphur burned. Table IV. gives details of the relative condensation in a number of series of chambers of varying construction and size. We have now some idea of the composition of the

attached to the sides of the chamber, and the results led him to conclude that Smith's conclusions were not trustworthy, and that on an average about 80 per cent. of the total acid produced was formed in the upper portion of the chamber within three or four feet from the roof. I carried out a long series of experiments upon a set of chambers, divided by curtains into six sections, as shown upon the plans, the curtains having their openings alternately at the top and bottom. These chambers were fitted up with a series of sampling apparatus, consisting of leaden trays exactly one foot square each, and communicating with the outside of the chambers by a lead pipe, enabling the quantity of acid to be measured at stated periods. These trays were supported at heights of 3ft. 9in., 7ft. 4in., and 11ft. 3in. above the bottom of the chamber, and there was also, in addition, the usual side drip pocket upon the chamber wall.

In the first series of experiments the trays were left open and uncovered, and the daily yield of acid which had condensed upon these trays

DIAGRAM 1.—SULPHUROUS ACID.

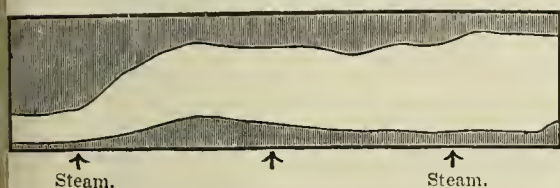


DIAGRAM 2.—SULPHURIC ACID.

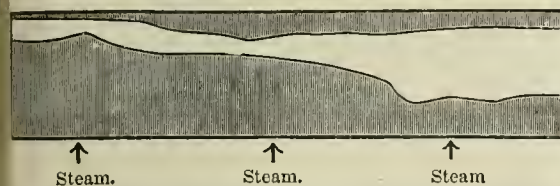


DIAGRAM 3.—NITRIC ACID.

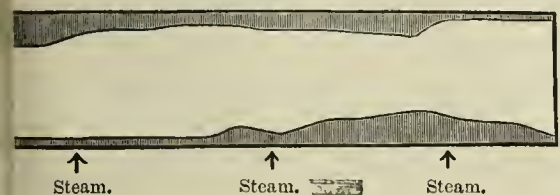


DIAGRAM 4.



gases in the various chambers of the series, and also the relative proportion of acid condensed in each chamber. We have now to consider the question as to zone or level of the chamber, in which the formation of the acid chiefly takes place. Mr. Smith concluded from his experimental results that the formation of acid was most active in the lower portion of the chamber, and from this cause he advocated the building of chambers of comparatively low height, but of considerable length. Mr. Smith's results, however, as shown by the figures which he gives, are somewhat misleading, owing to the method adopted for taking the sample, and his conclusion I believe to be quite an erroneous one. Some investigations have been made, I believe, by Mr. Hasenclever on the amount of acid formed upon trays suspended at various points in the chamber. I have not been able to obtain the details of these experiments, but Mr. Deacon, who also worked upon this subject, kindly gave me the results of his experience. His experiments were carried out by a series of gauges

was carefully recorded for some months, both as to quantity and strength. There was considerable irregularity observed, and this was found to be caused by the lead of the roof wrinkling, from its expansion and contraction, the acid condensing upon the roof lead dripping from these points or projections of the lead. Any change in the temperature of the air caused these wrinkles to alter their form, so that at one time a considerable quantity of acid might be formed and fall into the trays, while at another time this acid might be directed clear of them by one of these wrinkles of the lead. In order to avoid this source of irregularity I had covers arranged, hanging in such a way over the trays as to prevent any drip from the roof getting into them, and these covers were made also in such a way that they could be raised or lowered, as required. A set of results was obtained in the one case with the covers close to the roof of the chamber, and in the other with the covers lowered so as to include exactly one cubic foot of space between the cover and the tray. Taking the highest tray as



yielding 100 parts of acid, the amounts are shown in Table V., which were obtained in the same time by the middle and bottom trays. Reducing the figures in Table V. to the basis of 100 of OV made in each chamber, as found by the gauges, we have the averages shown in Table VI. The proportion of acid made is shown also in this table.

In the next series of experiments the trays were covered, the covers being kept close to the roof of the chamber, so as to leave free circulation of the gases over the trays. Table VII. gives the results. The quantity of acid made in each chamber is also given in this table.

Another series of experiments was made with the covers lowered, so that exactly one cubic foot of space was enclosed between the cover and the tray, with the view of determining the relation as to formation of acid in an equal chamber space at the different levels in the chamber. The results are given in Table IX. Reducing these figures to the basis of 100 parts OV in each chamber, as found by the gauges, as in the previous cases, we have the results found in Table X.

If we now take these various results, and arrange them side by side, as in the tables, we shall find that apparently the same amount of acid has been obtained from each set of trays. As each of the gauged trays had a different height or column of chamber gases between it and the roof, one would naturally expect that the lower trays would give an increased yield of acid corresponding to the amount of space above it, but this has not been so.

If we suppose the chambers to be divided into three zones or levels between (1) the roof and the top tray, (2) the top and the middle tray, (3) the middle and

TABLE XI.

Percentage of Acid made in each Tray.	19 Weeks' Average. No Covers Used.	Weekly Averages. Covers Used Close to Roof of Chamber.		Weekly Averages. Covers Used at 1 ft. above Trays.
		a	b	
Top tray ....	35	36	36	35
Middle tray..	32	31	33	32
Bottom tray..	33	33	31	33

bottom tray, and designate the yields obtained from these as A, B, C, thus we would have

Upper zone = A

Middle zone = A + B

Bottom zone = A + B + C

but as we have found that A is = A + B, and also = A + B + C, it is evident that B and C can have no value, and that, so far as these results are concerned, no acid has been formed in the middle and bottom zone. To try and make this result more clear, a number of analyses of the gases of one of the chambers was made, and the following figures were obtained :—

## TOP ZONE.

SO <sub>2</sub> grms. per litre.....	126 as SO <sub>2</sub>
SO <sub>2</sub> grms. per litre. Total.....	159 as SO <sub>2</sub>
Per cent. existing as SO <sub>3</sub> .....	21 of total
Per cent. oxygen after absorption of acids	10.7
Per cent. oxygen after oxidising SO <sub>2</sub> .....	9.0

## MIDDLE ZONE.

SO <sub>2</sub> grms. per litre.....	112 as SO <sub>2</sub>
SO <sub>2</sub> grms. per litre. Total.....	159 as SO <sub>2</sub>
Per cent. existing as SO <sub>3</sub> .....	26 of total
Per cent. oxygen after absorption of acids	10.9
Per cent. oxygen after oxidising SO <sub>2</sub> .....	9.3

## BOTTOM ZONE.

SO <sub>2</sub> grms. per litre.....	108 as SO <sub>2</sub>
SO <sub>2</sub> grms. per litre. Total.....	161 as SO <sub>2</sub>
Per cent. existing as SO <sub>3</sub> .....	33 of total
Per cent. oxygen after absorption of acids	10.9
Per cent. oxygen after oxidising SO <sub>2</sub> .....	9.3

Extracting these figures again, we have :—

	Total as SO <sub>2</sub>	as SO <sub>2</sub>	as SO <sub>3</sub>	% SO <sub>3</sub> made in
Top .....	100	100	100	63
Middle..	96	89	124	15
Bottom..	101	86	157	22
				100

and, as the final result, we find from these analyses 63 per cent. of the sulphuric acid actually formed has been in the top portion of this chamber. It is clear, therefore, that by far the larger proportion of the acid has thus been made in the upper portion of the chamber, while the figures in Table X. would indicate that all the sulphuric acid must have been produced within the space between the roof of the chamber and the top tray, or, say, one-fourth the total height of the chamber.

We are safe, therefore, in drawing the general conclusion that almost all the acid is produced in the higher portion of the chamber. That this is quite in accordance with what might be looked for, we have only to examine the relative specific gravity of the gases at 0° C., compared with air—

SO <sub>2</sub> .....	2.217
SO <sub>3</sub> .....	2.760
N <sub>2</sub> O <sub>2</sub> .....	1.039
Air .....	1.000
H <sub>2</sub> SO <sub>4</sub> .....	1.430.000
Water .....	773.000
NO .....	1.589
Steam (100 C).....	.47

and to consider that we have to deal with matter in two forms, gaseous and liquid, the latter being in the form of a mist or spray of sulphuric acid, which being so very much heavier than the gases with which it is mingled, rapidly falls towards the bottom of the chamber, displacing the gases and forcing them to the upper portion, where naturally their condensation must take place. The average specific gravity of the gaseous matter may be roughly taken as about 2.00, while the sulphuric acid H<sub>2</sub>SO<sub>4</sub> has a specific gravity of 1.430.00, more than sufficient to account for the relative positions in the chamber. The figures which I have obtained, no doubt, show certain irregularities in the yield obtained at the various points of the chamber, but this is, I think, accounted for sufficiently by the agitation and whirl of the gases, induced by the strong effect of the steam jets entering the chamber and the constant current flowing in and out of the chamber. To what practical conclusion, then, do these results lead us? I think simply to that of having ample chamber space, the form of the chamber not being so material. With low chambers the power of mixing the gases is deficient, and a scarcity of nitrous compounds at any time does more harm from the going through of the chambers, while in high chambers there is more chance of the gases being thoroughly intermingled, and this "going through" is much more rare. What form of chamber will give the best results is a difficult question to answer, as the question of space is complicated with the question of "surface condensation," which plays a very important part in the production of the acid. That this is so is easily seen by watching, through the open door of a chamber, the constant drop of acid formed upon the roof of the chamber. The use of increased condensed surface has been often proposed, and notably by Mr. Ward, who proposed using piles of glass sheets, separated from each other by about an inch of space, and placed in chambers. The effect of surface upon the production of sulphuric acid was also investigated, and a tray, as nearly as possible one square foot area, suspended in the chamber, was found to give a quantity of 708.75 grms. H<sub>2</sub>SO<sub>4</sub>. Placing now in the tray, in a vertical position 1 in. apart, 12 pieces of glass, each 12 in. by 6 in., the amount of acid obtained in 24 hours

was 1644grms.  $H_2SO_4$ . This would show the condensation due to the glass is  $= 1644 \div 708.75 = 935.25$ grms., and as the surface exposed was equal to 12 square feet, the acid condensed on the glass surface was equal to 79grms. per square foot, the glass being vertical. Placing now the glass horizontally and carefully at the same distance apart as before, the acid obtained in 24 hours amounted to 3,226grms.  $H_2SO_4$ , giving  $3226 \div 708.75 = 2517.25$ , this being equal to 209grms. per square foot. If we take the amount produced in the tray without glass as being equal to 100, the relative proportions will be—

Tray without glass .....	100
Tray with glass, vertical .....	231
Tray with glass, horizontal .....	455

This enormous increase in the condensation ( $4\frac{1}{2}$  times) indicates how important a factor surface available for

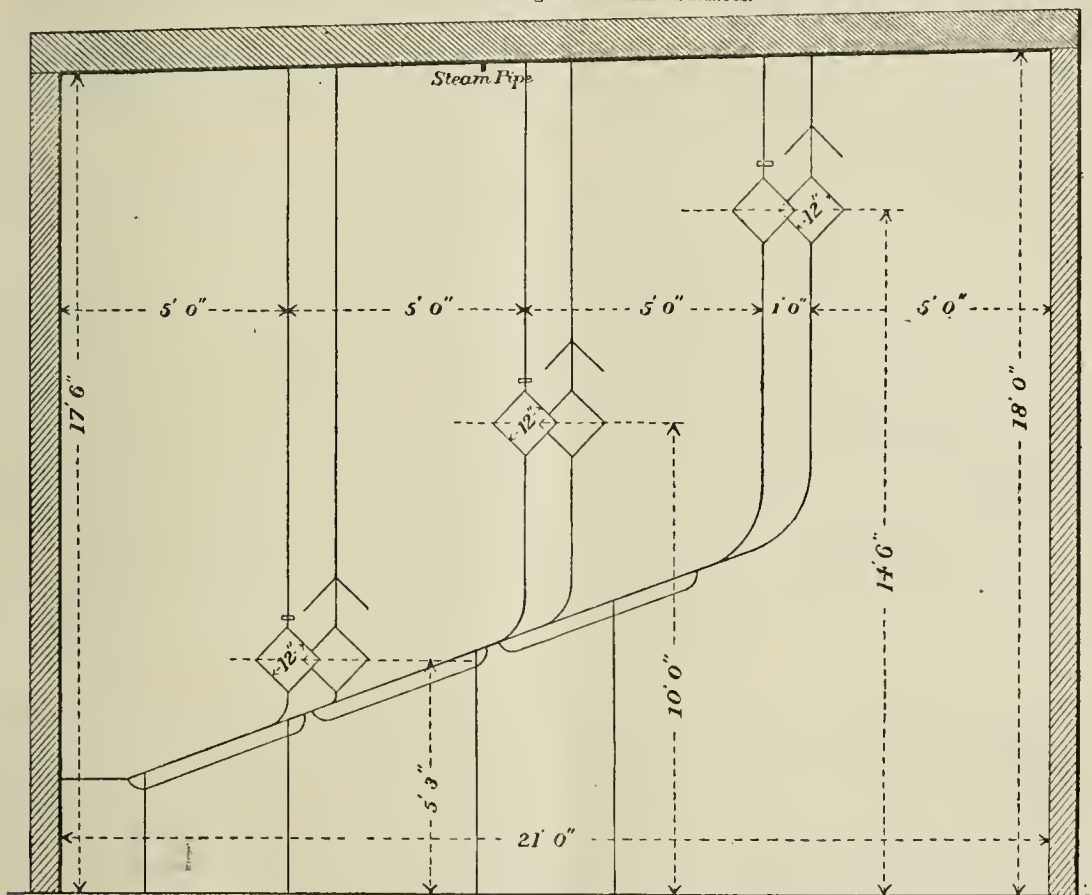
shield or cover over it, to prevent any acid from the upper portion of the chamber falling upon it, and these condensers were arranged at three heights, 5ft. 3in., 10ft., and 14ft. 6in. from the bottom of the chamber. The acid obtained by condensation upon this surface was measured at fixed hours, and the results tabulated daily. The observations were carried on for several months, and the average amounts obtained were—

For the Uncovered Trays.	Covered Trays.
High .... 100 OV ( $H_2SO_4$ ) .....	100 OV ( $H_2SO_4$ )
Middle .. 111 OV .....	111 OV
Bottom .. 165 OV .....	116 OV

representing as yield of—

High, say 61 % of total .....	63 % of total
Middle, say 8 % of total .....	7 % of total
Bottom, say 31 % of total .....	25 % of total

DRAWING A. Arrangement of Lead Surfaces.



condensation is in deciding upon the most advantageous form of chamber; but there has also to be considered in connection with the question the cost of erection. The chambers in the form of a cube would have the advantage of giving the maximum space with the minimum of lead; but in so far as surface condensation is concerned, it is clear that this would be the worst form of chamber that we could adopt. Some further experiments were made with a series of drips and trays, arranged as in the drawing marked A. These, which might be called surface condensers, were arranged in pairs, and means for collecting and measuring the acid condensed upon them were provided. One of each pair had a

confirming very completely our previous results, and further proving that the chief condensation took place in the upper portion of the chamber. In drawing B the arrangement of these "surface condensers" is shown in the first division. Six trays were arranged as in drawing A, each 1 foot square, and also a series of flat trays with surfaces of 1, 2, and 4 square feet respectively. These were used in order to find, if possible, the amount of condensation practically in proportion to the surface exposed, the figures being—on the 1 square foot, 100; on the 2 square feet, 199.2; on the 4 square feet, 403.

In the second division of the chamber the arrangements of the "surface condensers" were such that one

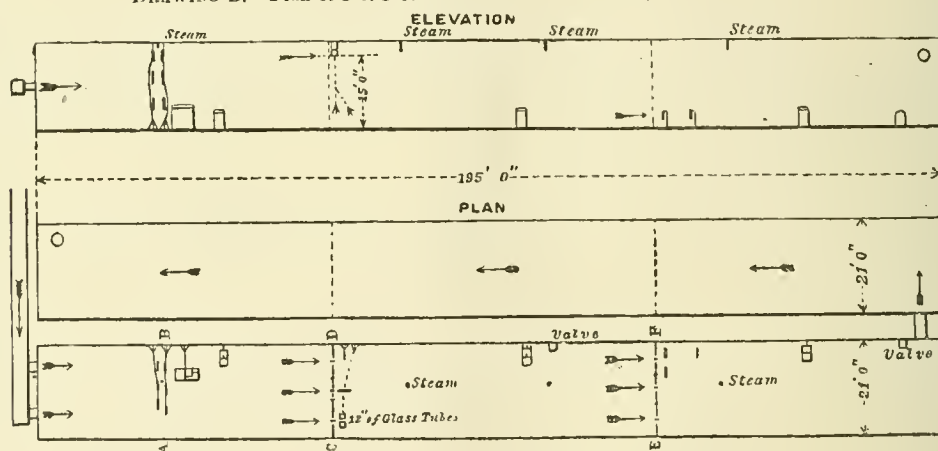


of them faced the current entering the chamber, and had its run-off pipes so arranged that the amount of acid formed on each side could be measured separately. The proportion obtained on the flat surface facing the current being taken as 100, the amount formed on the "lee" side, away from the current, was 78. Another "surface condenser" was placed with its edge towards the gas inlet, and the amount obtained from the double surface was 175, as compared with 178, in the previous cases. There being three openings in the curtains, opposite the third one was placed a pair of leaden trays of 1 square foot area. One of these had, in addition, slips of glass, 1 square foot area, placed in it vertically, and the amounts of acid obtained were in the proportion of 100 from the simple tray, and 192 from the tray with the extra 1 square foot of glass surface. These results show how very important, indeed, the question of surface is in connection with condensation, but the question of the best form of chamber is so involved with the question of cost, tear and wear from lead surface exposed, etc., that it would require a very large amount of calculation and experience before one could decide what form of chamber was the cheapest to erect, keep in repair, and give the best results as to

ticular leads, which are so pure and do not stand the action of sulphuric acid in the cold, are also the least suitable for chamber purposes, and whether the ordinary test which is applied for such lead—that is, merely ascertaining the amount of hydrogen evolved in a given time from a given surface—is also a sufficient test of lead for chamber purposes. That test has been objected to, on the ground that the conditions which exist in chambers are different. It would be very interesting to know whether the different portions of the chamber—that is, the lead at the different zones or regions of it—is acted upon differently according to the nature and composition of the gases at these particular points, and generally, whether the lead which behaves worst under sulphuric acid alone, is also most acted upon under chamber conditions.

Mr. BOYD (St. Rollox): As for the tests we use for "chemical lead," we have none of any great value. The hydrogen test was used for a considerable time, but we gave it up, as the results were misleading. Chamber lead is not so liable to be destroyed by the action of sulphuric acid as by the nitrous compounds. The action is different, and varies much with the position of the lead in the chamber. For

DRAWING B. Plan of No. 2 Series of Chambers.—Scale, 40 feet to one inch.



condensation and acid. I place these remarks before you merely as a contribution towards the solution of the problem, and I trust that the subject will be taken up by others and worked out still more fully.

#### DISCUSSION.

Mr. R. R. TATLOCK: I would like to ask as to the behaviour of the lead in the different zones or regions of the chamber. We have heard a great deal in our day about "pure chemical lead," but most of us know that this is now considered a contradiction in terms, and that pure lead is far from being well suited for many purposes connected with vitriol, particularly for the manufacture of the lead boxes in which vitriol is packed for export. Sometimes these are bulged to such an extent that the cube becomes almost a complete sphere on account of the action of the lead on the sulphuric acid and evolution of hydrogen. Sometimes in the lead pans also there is strong action, which seems to be due to the purity of the lead, because that lead, on analysis, shows usually mere traces of impurities. I should like very much to know if Mr. Boyd can tell us whether the par-

chambers and for concentrating pans we try to get as pure "virgin" lead as possible, containing its full amount of silver derived from the ore, and that has never been treated by any of the processes for desilverisation. But even the most carefully-selected sheets go wrong in the concentrating pans, and there is sometimes no accounting for it. A good deal depends on the workmen having temperature, strength, and supply of acid in good order.

Mr. STANFORD: I would simply rise to propose a vote of thanks to Mr. Mactear for the excellent paper he has given. It involves a considerable amount of labour, and gives us some indication of an attempt to solve that very important problem, "how to make the best vitriol chamber." It does seem a little remarkable, after all we know already, and all that Mr. Mactear has added to our knowledge of vitriol condensation and the great value of surface condensation, that we should still be obliged to use such large spaces to condense vitriol. The time is, I hope, coming when some smaller arrangements may answer the purpose. At present, I think Mr. Mactear has himself admitted that we have not yet arrived at even the proper shape of the vitriol chamber.

## Journal and Patent Literature.

## II.—FUEL, GAS, AND LIGHT.

*The Scheurer-Kestner Experiments on the Employment of Coal in Steam Boilers.* Ferd. Fischer. Dingl. Polyt. Jour. 251, 323.

SCHAUER-KESTNER used a narrow platinum tube (*vide Bulletin de Mulhouse* 1868, pp. 211 and 215) for taking the samples of gas, the slit of it being only a few tenths of a millimetre wide. A stream of gas was continuously aspirated through the tube for several hours by means of a water pump, 0.4 to 0.2 per cent. of the gas being sucked into a flask, by allowing mercury to flow out, for subsequent analysis. The difference in pressure between the mercury flask and the external air was 2mm. to 3mm. of mercury, but it rose as soon as the slit began to choke. As the action of the water air-pump could not be quite uniform, and the draught in the chimney was not steady, and the composition of the gases of combustion greatly varied (*Bulletin*, 1868, p. 337, and 1869, p. 280), it cannot be considered that a really average sample was obtained. The examination of the gases taken in this manner gave the following results:—

No. of the Experiment.	Carbonic Acid.	Carbonic Oxide.	Carbon Vapour.	Hydrogen.	Oxygen.	Nitrogen.	Excess of Air.	Highest Temperature of the Smoke Gases.	Coal burned on 1 sq. m. grate surface per hour.	Duration of Aspiration.
1	13.08	0.83	—	—	4.84	81.25	pr. et.	—	K	Hrs
2	7.73	0.41	—	—	11.42	80.41	—	—	45	2.5
3	14.03	0.91	—	—	2.82	82.21	—	—	—	3.5
4	13.80	—	0.86	1.06	5.53	78.75	—	—	—	4
5	8.62	—	0.14	0.53	10.83	79.88	—	—	45	8
6	13.21	—	0.52	1.08	5.27	79.92	—	—	—	2
7	8.23	—	0.04	0.52	11.35	79.86	—	94	16.6	3
8	12.89	—	0.23	0.96	5.53	80.34	—	93.2	23	3
9	14.63	0.86	0.49	0.56	2.80	80.66	—	13.32	126	47
10	10.87	—	0.19	0.19	8.99	79.76	—	42.84	156	92.5
11	14.16	0.97	0.98	1.11	2.18	80.60	—	10.47	128	47
12	14.87	0.84	1.15	1.35	1.41	80.38	—	6.66	119	40
13	13.34	—	0.46	0.91	3.77	81.52	—	17.61	135	40
14	13.13	0.24	0.32	1.41	4.42	80.18	—	20.94	—	40

The coal employed in these experiments had in round numbers the following composition:—

Carbon	70.0
Hydrogen	4.0
Oxygen	4.0
Nitrogen	1.0
Ash	21.0

100.0

Scheurer-Kestner concludes (*Bulletin*, 1868, p. 319) that the products of combustion contain the smallest quantity of combustible gases, when 10 cubic metres of air are supplied for 1 kilo. of coal, *i.e.*, about double the quantity theoretically necessary. This conclusion has given rise to the erroneous idea that twice the theoretical quantity of air must always be supplied, whereas F. Fischer has shown (*Dingl. Polyt. Jour.* 232, 345, and 250, 75) that it is better to admit less air. For estimating the loss in the smoke, samples were taken in the way suggested by Sainte Claire-Deville. A horizontal pipe was introduced into the heating flue, provided with openings on the top side, so that when water was made to flow through, the gases were sucked along with it and collected in a gasometer. Five samples taken in this manner had the following composition:—

	I.	II.	III.	IV.	V.
Nitrogen.....	87.18 ..	87.60 ..	86.60 ..	85.78 ..	84.31 ..
Carbonic acid ....	7.16 ..	5.27 ..	3.33 ..	5.63 ..	3.77 ..
Oxygen .....	4.66 ..	7.16 ..	9.86 ..	8.58 ..	11.92 ..

On the strength of the foregoing fourteen analyses and of six other analyses of smoke gases, Scheurer-Kestner assumes that the combustion gases contain 1 per cent. combustible gas, so that with 21 per cent. oxygen in the

air the actual composition of the gases may be accepted as under (*Bulletin*, 1868, p. 323, and 1869, p. 273):—

	I.	II.	III.	IV.	V.
Nitrogen.....	79.0	79.0	70.0	79.0	79.6
Combustible gases	1.0	1.0	1.0	1.0	1.0
Oxygen .....	4.1	7.3	9.6	7.8	11.2
Carbonic acid.....	15.9	12.7	10.4	12.2	8.8

On the basis of this bold assumption further calculations are made. The following experiment with Friedrichsthal coal may serve as an example (*Bulletin*, 1869, pp. 295 and 347):—

April.	Coal burned.	Water evaporated.	Amount of Oxygen in Gases.			Temperature.		
			Highest.	Lowest.	Mean.	Of the Water.	Of the Smoke.	Of the Air.
17	1063	6870	11.1	5.8	10.0	10.0	121	18
18	963	6000	9.1	6.1	8.0	9.8	108	20
21	1010	6250	10.2	5	8.1	10.0	113	19
24	918	6050	13	6.3	8.7	11.0	98	22
25	955	6500	13.5	9.3	10.7	11.0	111	24
				mean	9.1	10.5	110	20.6

From this it follows—

	Per cent.
Amount of oxygen found .....	9.1
Correction .....	2.3
Amount of oxygen.....	6.8

From this it is further concluded that the combustion gases contain 12.2 per cent. carbonic acid, and that for 1 kilo. coal, 10.715 cubic metres nitrogen and air, along with 1.485 cubic metre carbonic acid escaped. These correspond to a water value of 3.705 kilos., or at 101.4 excess temperature to 372 cal. It is further assumed from the 20 analyses that 5.5 per cent. carbon and 15 per cent. hydrogen escaped unconsumed, corresponding to 595 cal., that 1 per cent. carbon escaped in the smoke, corresponding to 63 cal., and that 282 cal. were carried away by the water vapour in the smoke gases, thus—

	Cal.
Steam .....	5013
Gases .....	372
Combustible gases .....	595
Soot .....	63
Water vapour.....	282
	6325

The calorimetric estimation of the average sample gave 8,457 cal., so that 2,132 cal., or 25.2 per cent., were lost by conduction and radiation. "The calculations of loss thus rest simply on the oxygen determinations in samples taken over water, and on assumptions and inferences of so bold a nature that serious criticism is disarmed." The writer considers it his duty to draw attention to the great defects in these boiler experiments, and to warn against the false conclusions which have been drawn from them.—W. M.

*Researches on the Use of Combustibles for Heating Steam Boilers.* Scheurer-Kestner. Bull. de la Soc. de Mulhouse, Nov.-Dec., 1883.

THIS is a reply to the different criticisms that have appeared on the results of some experiments undertaken by the author in conjunction with M. Meunier Dollfus, and published some fifteen years back. The plan followed was to determine the heat of combustion of the substance under examination in a Favre-Silbermann calorimeter, comparing the result with the value obtained for wood charcoal in the same apparatus. After this the substance was tried against wood charcoal under a steam boiler. The reply refers chiefly to the statements of M. Bunte, who has been commissioned by the Society of Munich to investigate the properties of the principal fuels



of South Germany. The author contends that the apparatus in which M. Bunte professed to determine the heat of combustion and the quantity of heat which may actually be utilised in practice by one and the same experiment was an imperfect calorimeter, and this assertion he supports by showing that M. Bunte has lost in the case of coke from 1.3 per cent. to 4.7 per cent. of the true calorific power. Moreover, he has never proved the accuracy of his apparatus by burning a substance such as wood charcoal, of which the heat of combustion is perfectly established—a precaution always adopted by M. Scheurer-Kestner. This is the more extraordinary, as M. Bunte himself discovered, after 29 experiments, that his results were, on his own showing, 14 per cent. too low, from heat lost by radiation, and he had to make some alterations in his apparatus to insure a more perfect conservation of the heat. One of the principal facts brought to light by M. Scheurer-Kestner, and most strongly attacked by M. Bunte, was that certain coals give a greater heat of combustion than is afforded by the addition of the heats of combustion of their hydrogen and carbon. The author shows, however, that M. Bunte has actually corroborated this in some of his researches, but in all cases he has drawn false conclusions from his results, by not taking into consideration the ash of the combustible. When this defect is remedied in the calculations his results are seen to be, on the whole, far too low to allow implicit confidence to be placed in them. M. Bunte appeals for support to the experiments made by M. Stohmann, of Munich, with a new apparatus (which is, in fact, only a modification of Frankland's calorimeter, given up by the inventor as inaccurate). As, however, this does not allow an estimation of the ash of the combustible used, and as no account has been taken of this ash, the results cannot be allowed to weigh with those obtained from a calorimeter of such known excellence as the Favre-Silbermann. In his experiments on the amount of heat which may actually be utilised in practice, the author came to the conclusion that about 25 per cent. is lost, and cannot be traced by known methods of investigation. M. Bunte thinks these figures far too high, but M. Scheurer-Kestner points out that a similar result was obtained when pure wood charcoal was used. M. Weinhold, referring to the analyses of the gas produced by combustion, prefers to collect the sample over water covered with oil, but the author thinks his own plan of using mercury is better. M. Fischer is somewhat hypercritical, complaining that the specific heat of a mixture of oxygen and nitrogen in the gases from the combustion is reckoned as the same as common air; the correction would make a difference of only six calories in 7218. His other remarks show a complete ignorance of the method in which the author's researches have been conducted.—A. R. D.

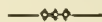
*Russian and Roumanian Petroleum.* Seifensied. Zeit.  
33, 395.

THE mercantile report of Tilsit contains the statement that the importation of American petroleum to Russia has wholly ceased. Russian petroleum is cheaper than American, and it is probable that in a few years Russia will not only produce sufficient for her own supply but will even export. Roumanian petroleum will probably become of importance for the supply of Eastern Europe, it being reported that an immense petroleum basin has been discovered. In Breslau, Russian petroleum of good quality from Baku is placed in the market 8 per cent. below the price of the American oil.—A. H. A.

*Testing Petroleum for Adulteration with Solar Oil.* Dr. G. Heppel. Chemisch-techn. Central-Anzeiger.

COMMERCIAL petroleum is said to have a density of from 0.795 to 0.804, solar oil from 0.830 to 0.870, so that a density of the sample higher than 0.804 points to the presence of solar oil. The boiling point of ordinary petroleum is said to range from 180° to 250° C., and that of solar oil between 240° and 300° C. In treating petroleum with concentrated sulphuric acid the oil remains colourless, and only the acid is coloured a light brown. If solar oil be thus treated, the sample becomes brown, and the acid a dark reddish-brown. On treating

a mixture of the two bodies with acid, the colour of the oil is the more intense in proportion to the quantity of solar oil present. After some hours, the red-brown colour of the oil changes to violet-red, whilst the acid remains brown. The foregoing method is not very satisfactory should it happen that the petroleum has been badly purified on the one hand and good solar oil has been used on the other. The author has devised a method which gives good results with American petroleum, but he has not yet examined other oils with it. The sample is treated with perfectly dry copper butyrate in powder. The salt will dissolve, giving a bluish-green colour to the solution, which is now heated. If solar oil be present a yellow colour is produced at 120° C., with separation of yellow flocks, the quantity being in proportion to the amount of the admixture. If this oil be absent, the oil remains green and clear, even at a temperature of 210° C. Upon cooling the solution, a yellowish-brown precipitate is at first thrown down, and upon this a second precipitate of a greenish-blue colour, the liquid above being a pale yellow. The first precipitate is due to the solar oil, the second to the petroleum. When the sample is unadulterated, the liquid above the precipitate due to petroleum, is almost colourless. The copper butyrate employed was prepared from ordinary commercial butyric acid.—A. H. A.



*Improvements in the Manufacture of Gas and Apparatus Employed Therein.* Sydney Pitt, Sutton. Eng. Pat. 2544, May 22, 1883.

By means of any kind of heating gas conducted into a chamber partly filled with lime, and burned with air under pressure, this lime is raised to an intense heat. Steam (preferably superheated) and naphtha, or some other liquid hydrocarbon, are then introduced. The resulting gaseous mixture, consisting mainly of hydrogen and carbonic acid, is passed through a cooler into a governing holder, which maintains a constant pressure upon the gas. The carbonic acid is next removed by a passage through chambers partly filled with cold quicklime, care being taken to have a certain quantity of moisture present. Almost pure hydrogen is now left, which may either be used as such or else first carburetted by being passed through a chamber filled with brick or other refractory material raised to a high temperature in the way described above, and into the top of which naphtha or its equivalent is introduced. This is converted into a fixed gas by the hot material, and mixing with the hydrogen passes away to the distributing holder. When the quicklime used for absorbing the carbonic acid has become spent, it may be regenerated by being heated in the same way as the lime and brick chambers above. By another portion of the invention an independent generator for the heating gas may be dispensed with by burning the hydrocarbon with air in the first lime chamber until the temperature is sufficiently raised, and then stopping the air supply and admitting steam. When the temperature falls again below the point of conversion air is again admitted, and a fresh combustion of the hydrocarbon reheats the apparatus.—A. R. D.

*Improvements in Gas Generating Furnaces.* L. Mond, Northwich. Eng. Pat. 3821, 1883.

THE inventor claims (1) the manufacture and use of gas generating furnaces having air-admission grates in the form of a long slit or slits, or grate or grates, or number of orifices near together, so as to accomplish the same result, extending nearly or quite the entire length of one, or preferably two, opposite sides of the producer chamber, and at a distance above the opening through which the ashes or clinkers are withdrawn, and (2) the manufacture and use of rectangular gas-generating furnaces, having two working fronts opposite each other, through each of which air, or air and steam, or air and other gases, can be admitted, and which allow the furnace to be clinkered from both sides. The advantage of this producer is that every part is very accessible when working. The air is admitted from the outside into grates almost incapable of being impeded by clinker,

the fuel can be got at by working bars from every side, and the cliniker can be cleaned out without in the smallest degree disturbing the working of the producer from either or both sides of its chamber. Thus, inferior kinds of fuel can be used, such as slack, shale, and other dirty fuel not fit for other gas producers.—D. B.

*Apparatus for the Manufacture and Treatment of Gas.*  
J. E. Dowson, London. Eng. Pat. 3406, July 10, 1883.

THE inventor surrounds a fire for generating heating gas with a double casing containing water. This double casing communicates at the bottom by means of pipes with a coil in which steam is produced, and superheated by a fire suitably applied. Part of the steam is conducted to the top of the space within the double casing, so that water is always forced into the coil under pressure, and part is conducted to an injector, whence it passes with air into the generating fire. Within the gasholder are two scrubbers, and the gas from the generator is conducted up through one into the holder, and has to pass down through the other to where wanted.—A. R. D.

*An Improved Regenerative Gaslighting Apparatus.*  
Anthony Spencer Bower, St. Neots, and Thomas Thorp, Whitefield, Lancaster. Eng. Pat. 3742, July 31, 1883.

THE gas is burnt from a ring burner, composed wholly or in part of steatite, having the necessary perforations in its periphery. The heated products of combustion are deflected towards the centre by a perforated annular curved plate, and pass upwards through a series of vertical tubes in a closed chamber (forming the "primary regenerator") to the chimney. The air necessary to combustion is introduced through and heated by the "secondary regenerator," which is formed by a number of plates attached alternately to the bottom plate of the primary regenerator, and to the upper side of the curved deflector; thence it passes, partly through the perforations of the deflector into the glass containing globe, but mainly into the primary regenerator; and thence through one tube surrounding the gas supply pipe to the interior of the flame, and through a second, passing within the gas pipe, to the exterior of the flame. "The secondary regenerator heats that portion of air admitted directly to the globe, and also partially heats the whole of the air supplied through the primary regenerator for combustion with the gas." A slight modification of the arrangement is made for use with table lamps.—W. G. M.

*Improvements in the Manufacture and Purification of Illuminating and other Gases, and in Apparatus used in such Manufacture and Purification.* Alexander Gibb Henderson, Wood Green, and James Anderson Kelman, High Holborn. Eng. Pat. 3708, July 30, 1883.

THIS invention relates first to a new form of washer scrubber. It consists of an upright case divided horizontally into eight compartments. The floor of each chamber is kept covered with water to a certain depth by means of overflow pipes, the water being constantly renewed from above, and the saturated water drawn off from the lowest chamber. The gas enters through the lowest division, thence passes upwards through bell-mouthed tubes, supplied with distributing caps, of which several are fitted through the floor of each chamber, and thus to an outlet in the uppermost compartment. These distributing caps serve to slightly water-seal the tubes, and are fitted interiorly with curved vertical divisions, which cause the gas to divide and give it a circular motion, inducing whirlpool currents in the water. Secondly, "to the condensation and partial purification of the gas preparatory to its passage to the water-scrubber." This is effected in three vertical multitubular vessels arranged side by side. Each vessel is provided with an open water chamber above and a closed one below, and with one or more perforated discs dividing it into horizontal compartments. The perforations are somewhat larger than is required for the vertical tubes to pass through, the area of the excess being at least equal to that of the gas supply pipe. The gas enters at the top of one of the chambers, is cooled by the water and the discs, leaves at the

bottom, and is passed to the top of the second chamber, finally leaving at the bottom of the third vessel. The water flows in a contrary direction; cold water is supplied to the closed chamber beneath that vessel, through which the gas passes last, rises through the tubes to the upper cistern, thence to the lower cistern of the vessel next in order, and so on, the circulation being maintained by the heating of the water. Thirdly, to the recovery from the liquid deposited in the hydraulic main of certain illuminating properties of which the gas has been deprived while passing through the main, and of restoring the same to the gas before passing through the condensers, and also to the partial purification of the gas so treated. The liquid from the hydraulic main is caused to trickle over perforated serpentine plates, through which the gas is passed several times, in a large upright chamber, thence the liquid flows over steam pipes in a lower compartment, giving up volatile hydrocarbons to the enrichment of the gas, and is finally removed by a trapped outlet pipe beneath. The gas passes in a contrary direction to the fluid.—W. G. M.

*Treating Spent Lime from Gasworks.* W. R. Lake, London. Eng. Pat. 3657, July 25, 1883.

THE spent lime is thoroughly leached with hot water or steam, and the resulting liquor treated in a closed vessel with a stream of carbonic acid gas. Sulphuretted hydrogen is disengaged and conducted away to a chamber, either to be absorbed by hydrated oxide of iron or to be burned for the manufacture of sulphuric acid. Sulphocyanide of lime is left in the closed vessel, and this is decomposed by the addition of sulphate of potash. The liquor is separated from the precipitated sulphate of lime, and the salt yielded by its evaporation is used in the manufacture of ferrocyanides.—A. R. D.

*Improvements in the Manufacture of Hydrogen Gas.*  
Sydney Pitt, Sutton. Communicated by Erazm. J. Jermianowski, New York, U.S.A. Eng. Pat. 5709, Dec. 11, 1883.

A MIXTURE of hydrocarbon vapour and steam is passed successively through two chambers containing respectively lime and anthracite (previously heated by the combustion of hydrocarbon vapour within them). The carbonic acid, which, together with hydrogen, is the product from the first chamber, is thus converted by the red-hot anthracite into carbonic oxide; the resulting mixture of hydrogen and carbonic oxide, after washing, is ready for use. Or the water gas may be prepared in another way. Two or more cupolas filled with anthracite, and provided with a pair of air regenerators, are connected with an ordinary gas producer. The gases from the latter are employed to heat the anthracite in the first cupola, and afterwards one of the regenerators; they are then diverted to the second pair, the air used for the combustion of the gas in the second cupola being heated in the first regenerator. Meanwhile a current of steam is passed through the first cupola, thereby yielding the required gas until the temperature of the anthracite has fallen too low, when the producer gas is again passed through this cupola, and the steam is allowed to act upon the now heated anthracite in the second generator. Thus a constant stream of water gas is obtained, which may be washed and stored for use.—W. G. M.

*The Purification of Coal Gas.* Miles Williams, Wigan. Eng. Pat. 3642, July 4, 1883.

IN order to save the nuisance of removing the oxide of iron from the purifiers, and of exposing it to the air for revivification, atmospheric air is drawn into the main (before the gas reaches the purifiers) in such proportions as to revivify the oxide. This air is first charged with the vapours of petroleum or some other hydrocarbon—but not gas tar—so that the illuminating power of the gas is not decreased. By this means the oxide can be left in the purifiers until it is so charged with sulphur as to be of no further use for the purpose.—A. R. D.



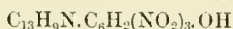
*Apparatus for Carburetting Gas and Air for Lighting and Heating Purposes.* John Thomas, London. Eng. Pat. 3359, July 6, 1883.

A RESERVOIR containing the hydrocarbon to be used for carburetting is connected by a pipe with a smaller vessel containing asbestos cloth, some of which passes down the connecting pipe and dips into the hydrocarbon, so that this latter passes into the smaller vessel by capillary attraction. The mixture of gas and air is first heated at the burner, and then passed through the smaller vessel. For heating, a larger proportion of air is required than for lighting purposes.—A. R. D.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*On Acridine Picrate.* R. Anschütz. Berl. Ber. 17, 438.

IN an investigation of the hydrocarbons present in crude phenanthrene the author treated the substance with an alcoholic solution of picric acid, and obtained a small quantity of a picrate, which was very slightly soluble even in boiling alcohol. An examination and analysis of the substance showed that it was acridine picrate



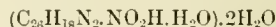
By rapid crystallisation from alcohol acridine picrate forms microscopic prismatic needles; by slow crystallisation it forms hard prisms grouped in stars. The salt possesses a canary yellow colour and melts above  $208^\circ$ , at which temperature it begins to blacken. It dissolves with great difficulty in cold water and is partially decomposed by boiling water, the smell of acridine being easily recognised. Acridine picrate is also very slightly soluble in cold alcohol and benzene, and the solubility is not much increased by boiling. Acridine may therefore be frequently employed in the analysis of picrates, the hydrochloride, for instance, in the analysis of picrates, and the free base in that of the picric acid compounds of hydrocarbons. In order to avoid the difficulty of washing the precipitate thoroughly with benzene when on a paper filter, the author recommends the employment of a platinum crucible the bottom of which is perforated and covered with asbestos. Many of the acridine bases appear to form difficultly soluble picrates. Thus the chrysanine compound is a brick-red powder which is almost insoluble in benzene. It is possible that a more insoluble picrate than that of acridine may be found, but this substance appears to give as good results as ammonia in the analysis of picric acid.—S. Y.

*II. On High-boiling Phenols contained in Coal-Tar.* Communications by E. Noetting. Berl. Ber. 17, 385.

IT is well known that phenols are contained in the high-boiling portions of coal tar, but their nature has not been studied. Some years ago the author obtained colouring matters by combining the phenols with diazo compounds. Thus sulphanilic acid gave a yellow-brown, and naphthionic acid a red-brown colouring matter, which, however, were not to be compared with those prepared from naphthol, etc. In order to study the nature of the phenols, the raw product, obtained by treating the tar oils with caustic soda and precipitating with an acid, was fractionated *in vacuo*, and the portion above  $300^\circ$  investigated. The crude products were obtained from Messrs. Vale and Company, in Hamburg, and Messrs. Dittler and Company in Höchst. The removal of the hydrocarbons still present in the distillate was effected by neutralising with caustic soda, diluting with a large quantity of water to precipitate smeary impurities, and extracting the hydrocarbons by agitation with toluene. The phenols were precipitated with an acid, extracted with ether, and distilled with zinc dust, and the semi-fluid mixture of hydrocarbons was at once oxidised with chromic acid. Small quantities of phenanthrene quinone and anthraquinone were found among the oxidation products. Hence it is probable that the phenols of phenanthrene and anthracene are present in the tar oil, but it is certain that a great number of other phenols are also contained in it.—S. Y.

*On Acridine.* L. Medicus. Ber. 17, 196.

THE recent adjustment of the formula for acridine induced the author to resume his former investigations on a nitroso-compound of acridine. On treating a solution of a salt of acridine with a solution of an alkaline nitrite, a light yellow flocculent, precipitate is produced, which, when crystallised from hot water, forms long, yellow, silky needles, melting at  $150^\circ$ – $151^\circ$ . The compound is sparingly soluble in cold water and ether, dissolves in hot water, and is readily soluble in alcohol. Its solutions exhibit the fluorescence which characterises the salts of acridine. Analysis gave numbers corresponding to the formula



The author found that by the action of zinc and hydrochloric acid on this compound acridine is reproduced. D. B.

*Hydrocarbons and Alcohols obtained from American Petroleum.* G. Lemoine. Ber. 17, 132, and Bull. Soc. Chim. 41, 161.

THE hydrocarbons, octyl-, nonyl- and decyl-hydrides obtained from petroleum by fractional distillation were separated from the unsaturated hydrocarbons by treatment with bromine. Octyl-hydride has a specific gravity of 0.732 at  $12.1^\circ$ , and boils at  $121^\circ$  at a pressure of 779mm., at  $82^\circ$  at 212mm., at  $63^\circ$  at 100mm., and at  $31^\circ$  at 27mm. Nonyl-hydride: (1) Boiling point  $135$ – $137^\circ$ ; specific gravity 0.742 at  $12.4^\circ$ . (2) Boiling point  $129.5$ – $131.5^\circ$  at 751mm.,  $59$ – $60^\circ$  at 65mm.,  $37.2$ – $40^\circ$  at 22mm.; specific gravity 0.743 at  $0^\circ$ , 0.734 at  $12.7^\circ$ , 0.731 at  $16^\circ$ , and 0.725 at  $24.7^\circ$ . Nonyl-chloride was prepared from a nonyl-hydride boiling at  $132^\circ$  at a pressure of 759mm. It had a specific gravity of 0.911 at  $23^\circ$ , 0.908 at  $25.8^\circ$ ; boiling point  $180$ – $184^\circ$ . Nonyl-alcohol obtained from nonyl-hydride, boiling at  $130$ – $135^\circ$ , had a specific gravity of 0.855 at  $18.5^\circ$ , and boiled at  $133$ – $136^\circ$ . Decyl-hydride: Specific gravity 0.764 at  $0^\circ$ , 0.753 at  $15.6^\circ$ , 0.751 at  $17^\circ$ , 0.739 at  $33.5^\circ$ ; boiling point  $151$ – $160^\circ$  at 757mm.,  $67.5^\circ$  at 36mm. Decyl-chloride: Specific gravity 0.908 at  $190^\circ$ ; boiling point  $201$ – $203^\circ$ . Decylene: Specific gravity 0.855 at  $14^\circ$ ; boiling point  $159$ – $174^\circ$ . Decyl-alcohol: Specific gravity 0.858 at  $18.5^\circ$ ; boiling point  $200^\circ$ .—D. B.

*Note on (1) the Behaviour of the Nitrogen of Coal during Destructive Distillation, and (2) a Comparison of the Amounts of Nitrogen left in Cokes of Various Origin.* By Watson Smith. Journ. Chem. Soc. April, 1884.

MR. WM. FOSTER, at the close of his interesting paper read before the Chemical Society, and published in its Journal (1883, Trans. 105–110), summarises his results as follows:—

Percentage of nitrogen in the coal .....	1.730
Percentage of nitrogen of coal evolved as ammonia during destructive distillation ..	0.251
Percentage of nitrogen in coal evolved as cyanogen during destructive distillation ..	0.027
Amount of nitrogen remaining in coke furnished by 100 parts of coal .....	0.842
	1.120
Nitrogen from 100 parts of coal unaccounted for, the larger portion of which is presumably present as free nitrogen in the coal-gas. Some is no doubt in the tar .....	0.610

If we further reduce these figures to percentages on 100 parts of nitrogen, their relation to each other becomes more intelligible:—

Nitrogen of coal evolved as ammonia .....	14.50
Nitrogen of coal evolved as cyanogen .....	1.56
Nitrogen of coal present in the coal gas in the elementary condition .....	35.26
Nitrogen of coal remaining behind in the coke .....	48.68
	100.00

"I have not made any experiments on the amount of nitrogen in the tar, nor am I in possession of any information on the subject, but I have assumed that the quantity is relatively small." Mr. Foster's last remark referring to the amount of nitrogen in the tar interested the author on first reading it, and for the following reason: As far back as the year 1868, when making distillations either

of coal tar or crude coal-tar oils, he observed, whenever the distillations were carried so far that pitch remained in the retort, and these residues of pitch were further heated until partial coking took place, that on leaving the whole to cool partially, and then removing the stopper of the retort, a smell of ammonia was always perceptible, reddened litmus paper was turned blue, and a stopper moistened with hydrochloric acid fumed strongly. Certain tar distillers aver that after coking pitch, when the manhole lids of the stills have been removed, the odour of ammonia is very powerful. The experiment is so easily performed with a little hard pitch broken up small and placed in a retort and heated to coking, that it would form a very good lecture experiment, to illustrate the fact that nitrogen compounds of considerable stability exist in hard pitch and in partially-coked pitch, and that at temperatures near the softening point of glass these compounds are so decomposed that on partial cooling and in contact with moist air ammonia is evolved. It is a remarkable fact that not the slightest smell of ammonia could be perceived, if the retort-stopper were removed whilst the retort is still very hot. The most favourable time for obtaining the evidence of the presence of ammonia is when the retort has cooled so far that its bulb may be held in the hand. The author supposes that the nitrogen in the partially-coked pitch has been brought into so unstable a form of combination, that, on cooling down and a back-suction of moist air into the retort taking place, such unstable nitrogen compounds in the highly-porous mass in presence of moisture are decomposed or altered so as to yield ammonia. It was determined to attempt to estimate the amount of nitrogen in ordinary coal tar and in the oils obtained at different stages from that tar, as well as in the pitch left as a residue. The numbers given are the means of at least two experiments for each sample. The tar analysed was thoroughly well settled from ammoniacal water, but beyond this no attempt was made to remove suspended solution of ammoniacal salts. The crude benzene contained ammonia in solution, which communicated to it a pungent odour. The "light oil" likewise smelt slightly of ammonia, but the "creosote oil" was quite free from ammoniacal odour. *Nitrogen in the Tar:* The mean of two experiments gave 1.667 per cent.

Crude benzene, obtained from the above tar (mean of two experiments) .....	= 2.327
"Light oil," obtained from the above tar (mean of two experiments) .....	= 2.186
"Creosote oil," obtained from the above tar (mean of two experiments) .....	= 2.005
Red oil, or oils from which the crude anthracene had been separated by filtration (mean of two experiments) .....	= 2.194
Pitch, obtained from the above tar (mean of two experiments) .....	= 1.595

It would thus appear that nitrogen compounds of high boiling points occur in the oils drained from the crude anthracene to a considerable extent, and that though there is a falling off in the pitch (in the above case a hard pitch), yet still in this product certain very stable nitrogen compounds occur; further, that the amount of nitrogen contained in ordinary coal-tar oils (from Lancashire coal tars) may be stated as 2 per cent. on the average. Of course the lighter naphthas will contain the pyridine bases, the intermediate naphthas and oils, more especially the quinoline bases, whilst the anthracene oils and pitch will contain the carbazols and acridine, and possibly other nitrogenous bodies of high boiling and melting points not yet examined. *Comparison of the Amounts of Nitrogen left in Cokes of Various Origin:* Three varieties of coke were now analysed, with the object of determining the nitrogen in them, viz., ordinary gas-retort coke, Beehive metallurgical coke, the hard compact metallurgical coke from the Simon-Carvés coke-ovens. The results are as follows:—

Kind of coke examined.	Percentage of nitrogen found.
(a) Ordinary gas-retort (mean of two experiments) .....	1.375
(b) Beehive metallurgical coke (mean of two experiments) .....	0.511
(c) Metallurgical coke from the Simon-Carvés coke-ovens mean of three experiments) .....	0.384

From the above results it may be concluded that in (a) the effect of a sharp heat of short duration is shown, under the usual circumstances of gas retort work, not to be so efficacious in decomposing certain very stable nitrogen compounds so as to drive the nitrogen out of the coke, as a long-continued high temperature such as is found in (c). In (a) a charge of from 2 to 3 cwt. of coal is heated for 6 hours, whereas in (c) a charge of 4 tons is heated for about 40 hours. The temperatures employed are not much unlike in (a) and (c), though on the whole the heat of the Simon-Carvés oven will be the stronger. No note was taken or inquiry made as to the *part* of the interior of the ovens from which the cokes under examination were taken. In the case of gas-retort and Simon-Carvés oven cokes no such note or inquiry is necessary, since in both cases external heat is uniformly applied to the mass of fuel disposed in a thin layer, which in the former case is arranged horizontally, in the latter vertically. With the Beehive coke, certain differences of temperature might occur in different parts of the mass, e.g., the portions of coke lying nearer the surface of the mass in the ordinary Beehive, and nearer the small air inlet, but not necessarily nearer the walls (not externally heated, but rather cooled), might be expected to have been more strongly heated than those lying next the floor of the oven. In the Jameson oven the temperature throughout the coking and coked mass will be much more uniform than in the ordinary Beehive. In the above-mentioned experiments, simply a well-burned sample of Beehive coke was taken for examination, exhibiting an average appearance and texture.—W. S.

*On the Examination of Petroleum.* Dingl. Polyt. Journ. 250 [4], 169-172. See this Journal III. [3], pages 161-162.

BEILSTEIN recommends the following process for testing the flashing point of petroleum (Fig. 1). A cylindrical

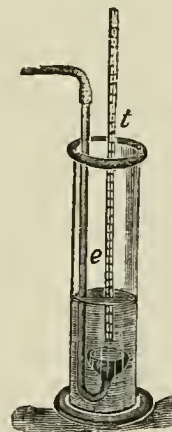


Fig. 1.

glass vessel of 35mm. diameter and 175mm length has two marks, one 60mm. from the bottom, the other 70mm. from the bottom. Petroleum oil is poured into the vessel till its level reaches the first mark. Air is conducted from a gasometer by means of an indiarubber ball-syringe attached to a fine brass tube *c*, terminating in a rose perforated with fine holes. The bulb of the thermometer *t* reaches to the middle of the oil. The apparatus is placed in a water bath, the temperature of which rises slowly 1° in from 2 to 3 minutes. Each time, when the temperature of the petroleum has increased about 1°, air is passed through for 5 seconds, and so quickly that the foam reaches up to the second mark on the glass vessel. At the same time a small flame is held at the mouth of the apparatus. (See pp. 161-162 of this Journal). The apparatus (Fig. 2) of Alex. Ehrenberg, of Dresden, is constructed on the syringe principle, and consists of



two limbs, A and B, combined together at E. The one contains the piston and the other the thermometer C. From the stem B a tube D leads to the ignition flame *a*. For use the apparatus is filled with the petroleum to be tested, and warmed or cooled down in the water bath till the

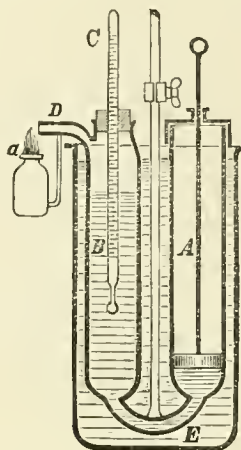
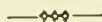


Fig. 2.

thermometer C indicates the experimental temperature. Then the piston is forced down, and thus a portion of the oil gases are carried to the flame. After again raising the piston rod, the experiment can be repeated until the flashing point be discovered.—W. S.



*Obtaining Ammonia, Tarry Matters, and Combustible Gases from Coal.* L. Mond, Northwich. Eng. Pat. 3923, August 13, 1883.

THE inventor has found that in the combustion of coal in gas producers a low and not a high temperature, as generally supposed, is most conducive to the formation of ammonia from the nitrogen contained in the coal. Nearly the whole of the nitrogen contained in the coal is obtained as ammonia, if the fuel be burned in the presence of steam at a temperature below the point of dissociation. To effect this a limited supply of air, loaded with water spray or steam in large quantity is introduced into the furnace. The resulting gases are richer in hydrogen and have a higher heating power, and the tarry matter is also richer and larger in quantity. The temperature may be also kept low by the use of air partly deprived of oxygen, *e.g.*, furnace gases. The ammonia and tar are collected by cooling and condensing, or the ammonia may be absorbed without cooling by a solution of calcium chloride or magnesium chloride, or by sulphuric acid. The point of novelty claimed is the employment of a low temperature of combustion by loading or charging the limited supply of air with so large an excess of steam or water that the temperature of combustion does not exceed a dull red heat.—C.C.H.

*Improvements in the Mode of Refining Paraffin.* C. Crellin, Leytonstone. Eng. Pat. 3717, 1883.

THE paraffin is melted and treated several times with sulphuric acid. It is then separated and mixed with fuller's earth at a temperature of about 180° F. This treatment is repeated at a temperature of 220° F., when the separated paraffin will be a lightly-refined wax.—A. H. A.

*Process of and Apparatus for Distilling Peat and Obtaining By-products therefrom.* F. C. Glaser, Berlin. Eng. Pat. 3855, 1883.

THIS invention has received provisional protection only. It relates to a process by means of which combustible matter in peat is converted into gaseous fuel for heating purposes, whilst at the same time the by-products, such

as ammonia, pyroligneous acid, tar, and hot air are obtained therefrom much more effectually than hitherto. The essential feature of success consists in so carrying out the operations as to carefully avoid the attainment of any high degree of heat.—D. B.

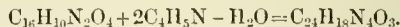
*Improvements in the Treatment of Bituminous Shales largely impregnated with Sulphur, and in obtaining Products therefrom.* C. Maclaren Irvine, Blackwood, and R. Slater, London. Eng. Pat. 3725, 1883.

THIS invention has for its object the distillation of shales containing large quantities of sulphur, the utilisation of the sulphuretted compounds given off during the distillation, and the prevention of nuisance. The shales are distilled at a low heat, with or without the application of steam, the watery and oily distillates being condensed in the usual manner, whilst the gases generated, consisting of hydrocarbons and sulphur compounds, are treated as follows: They are either simply burnt, and the resulting sulphurous anhydride is used for the manufacture of sulphuric acid, or sulphurous acid in a gaseous state, or in solution in water, is mixed with the gases or with sulphurous acid gas and steam, the result being the formation of water and elimination of sulphur. By adopting the latter method the gaseous hydrocarbons are at the same time purified, and can be utilised for heating or lighting purposes. The inventors also employ oxides of iron such as are used for the purification of coal gas, the resulting product being employed for the manufacture of sulphuric acid.—D. B.

#### IV.—COLOURING MATTERS AND DYES.

*On a Blue Colouring Matter from Pyrrol.* G. L. Ciamician and P. Silber. Berl. Ber. 17, 142.

THE discovery by Victor Meyer of the formation of a blue colouring matter by the action of isatin on thiophene, and the analogous behaviour of pyromucic acid, induced the authors to ascertain whether pyrrol would give a similar reaction. The colouring matter obtained by them appeared to be formed not like that of indophenin from isatin and thiophene, but by the union of a double molecule of isatin with two molecules of pyrrol, with liberation of one molecule of water, thus—



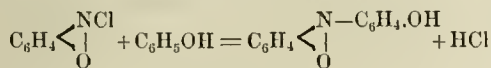
Two parts of isatin were dissolved in 50 parts of glacial acetic acid—pyrrol being partly decomposed by mineral acids—and 1 part of pyrrol was added to the boiling liquid. The liquid became dark blue at once, and was poured into water. The colouring matter was precipitated by the addition of sodium carbonate until the solution was nearly neutral, and the fine dark blue powder was filtered. After washing repeatedly with water, and drying, it was recrystallised from hot glacial acetic acid and then from alcohol. The crystalline structure of the dark blue powder was seen under the microscope. The analyses of the substance agreed fairly well with the formula  $C_{24}H_{18}N_4O_3$ , but the authors are not sure of its correctness, and are attempting to obtain confirmation of it. The colouring matter is soluble in glacial acetic acid, phenol, boiling alcohol, and concentrated sulphuric acid, but the latter solution becomes discoloured and deposits a black powder. The colouring matter is insoluble in ether. Its solution in acetic acid is decolourised by zinc dust.—S. Y.

*On Indophenols and Colouring Matters resembling Indophenols.* Richard Möhlau. Berl. Ber. 16, 2843.

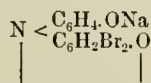
THE beautiful brown colouring matters obtained by Hirsch, Schmitt and Andresen, Witt, and others, which contain the common trivalent group  $>N-O-$  may be divided into two groups, those which resemble indophenols, and indophenols. According to the author the term "indophenol" should be applied to the former class of substances, since they possess the properties of phenols; for the latter he proposes the name "indoaniles," because they are formed from derivatives of aniline.

1. COLOURING MATTERS RESEMBLING INDOPHENOLS.

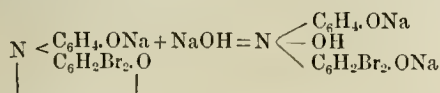
(*Indophenols*): These relatively unstable compounds may be obtained by four methods: (a) By the action of quinone chlorimides on alkaline solutions of certain phenols at the ordinary temperature, whereby blue alkali salts are formed which dissolve in alcohol with a fuchsin-red colour. (b) By oxidation of an alkaline solution of equal molecules of a paramidophenol and a phenol. (c) By the action of quinone chlorimides on phenols between 20° and 100°, when the substances corresponding to the blue alkali salts are formed. (d) By the oxidation of a solution of equal molecules of a paramidophenol and a phenol in acetic acid, by means of chromic acid. According to Hirsch the action of a quinone chlorimide on a phenol may be represented by the equation



but he did not succeed in obtaining conclusive proof of its correctness. The fact that the tendency of a substance to crystallise is increased by the introduction of electro-negative atoms or groups rendered it probable that more stable substances might be prepared by substituting chlorinated or brominated chlorimides for the quinone chlorimides, and the author therefore arranged with Herr Hirsch to investigate the action of dibromoquinone chlorimide on phenols. *Dibromoquinone Chlorimide*: By the reduction of dibromopara-nitrophenol with tin and hydrochloric acid the tin double salt of dibromopara-midophenol hydrochloride was obtained in colourless prismatic needles, and after recrystallisation from dilute hydrochloric acid was added gradually to an acidified 10 per cent. aqueous solution of calcium hypochlorite, whereby the double salt of dibromoquinonechlorimide was obtained almost quantitatively in the form of clear yellow or flesh coloured crystals. These were recrystallised from glacial acetic acid, and were found to melt at 80° and to decompose at 121°. The formation of the sodium salt of dibromoquinone phenolimide was best effected by introducing gradually and with agitation 5grms. of the chlorimide, triturated with dilute alcohol, into 8c.c. to 12c.c. of an alkaline solution of phenol containing 2grms. phenol to the cubic centimetre. The blue colouring matter was at once formed, and was deposited in gold-green glittering crystals, which were collected on the filter and washed with a semi-saturated solution of common salt. The aqueous solution of the crude product, saturated at 80°, was filtered, treated with one-third of its volume of caustic soda of specific gravity 1.29, and then with so much concentrated solution of tartaric acid that the blue colour just disappeared on warming. It was then quickly filtered, and exposed to the air in shallow vessels. The long prisms obtained in this manner appear gold-green by reflected and blue by transmitted light. They dissolve in water and alcohol with a pure blue colour. The formula

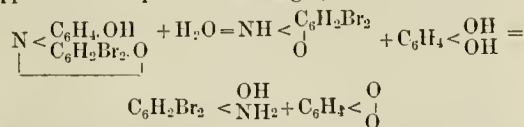


was confirmed by analysis. The blue colour of the solution disappears on warming with dilute caustic soda, but reappears on standing in contact with the air. Since neither the formation of a leuco compound nor the decomposition of the substance into dibromamidophenol and phenol can take place, the author offers an explanation of the reaction by assuming the formation of a substituted hydroxylamine, thus—

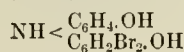


Dibromoquinone phenolimide is best obtained from the sodium salt by the gradual addition of acetic acid to its aqueous solution at 60°. It crystallises in deep red, almost black, prisms, which possess a metallic lustre. The substance dissolves in alcohol, ether, and glacial acetic acid with a fuchsin-red colour. It is insoluble in water. Concentrated mineral acids decompose the substance with for-

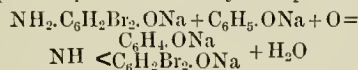
mation of dibromopara-amidophenol and quinone. This appears to take place in two stages, thus—



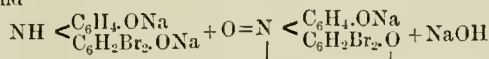
*Leucodibromoquinone Phenolimide*: The sodium salt obtained by the action of tartaric acid on the alkaline solution is very soluble and easily oxidised. It is therefore difficult to isolate, but the leuco compound itself is easily prepared in the form of colourless prisms by passing a current of sulphur dioxide into a warm aqueous solution of the sodium salt. It dissolves in the ordinary solvents with the exception of water, and melts at 170°, becoming at the same time dark coloured. It remains unaltered in dry air. The formula



was confirmed by analysis. It appears that the hydrogen atom of the phenol which occupies the para position with regard to the hydroxyl is eliminated by the chlorine of the chlorimide, so that the hydroxyl and nitrogen take the para position in the molecule of the dibromoquinone phenolimide. This view is supported both by the action of mineral acids on the substance and by the various colour reactions which are observed when the dibromoquinone chlorimide is treated with alkaline solutions of other phenols. Of the products thus obtained the majority may be crystallised. The identity of the substances obtained by methods (a) and (b), described above, was first proved by Köchlin and Witt (D. R.-P., No. 15915). In this case the reduction product of dibromoquinone chlorimide, dibromamidophenol, is employed, and the oxidation is effected by means of potassium bichromate or ferricyanide. The formation of dibromoquinone phenolimide may be represented thus—

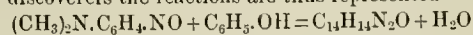


and

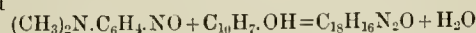


A solution of 26grms. dibromamidophenol and 10grms. phenol in 40grms. caustic soda of specific gravity 1.29 and about the same volume of water absorbs oxygen from the air with formation of the colouring matter. The oxidation is best effected with potassium bichromate at the ordinary temperature. The action is allowed to proceed until phenol can no longer be recognised. The substance is purified as described above. The methods (c) and (d) are not available in practice, for the decomposition of the substance occurs simultaneously with its formation.

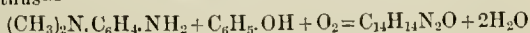
2. *INDOPHENOLS*.—*Indoaniles*: These blue colouring matters differ from those described under (1), in their inability to form soluble salts with ammonia and alkalis, and especially in that they exhibit basic properties, due to the presence in the molecule of the tertiary amido group  $\text{N}(\text{CH}_3)_2$ . Two representatives of the group, phenol blue and  $\alpha$ -naphthol-blue, are described in the patent to which reference has been made. Both substances are obtained by two methods. (a) By the action of nitrosodimethylaniline on an alkaline solution of phenol and of  $\alpha$ -naphthol, especially in presence of reducing agents such as zinc dust or tartaric acid. According to the discoverers the reactions are thus represented—



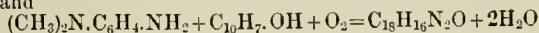
and



(b) By the oxidation of an alkaline solution of equal molecules of dimethylparaphenylenediamine, and of phenol and  $\alpha$ -naphthol respectively at the ordinary temperature, thus—



and

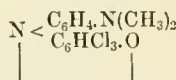




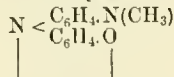
Trichloroquinonedimethylanilenimide, prepared by Schmitt and Andresen about the same time that Köchlin and Witt discovered the indophenols, is formed by the action of trichloroquinonechlorimide on dimethylaniline, thus—



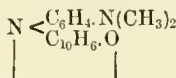
These three substances resemble each other in their stability in presence of alkalis in the cold, and in the ease with which they are decomposed by concentrated acids. The structural formula of the trichloro compound is certainly



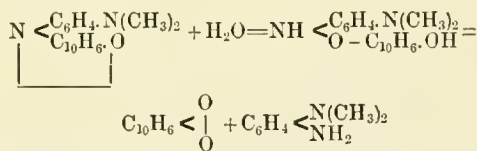
and it may be derived from phenol blue, which would then be benzoquinonedimethylanilenimide



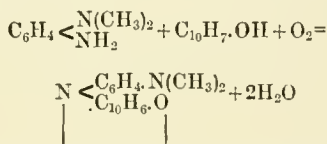
while  $\alpha$ -naphthol blue would be  $\alpha$ -naphthoquinonedimethylanilenimide



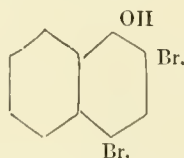
Although no direct proof of the constitution of these compounds has been obtained by the author, the following observations support this view of their structure: In the specification of the patent it is stated that the indophenols dissolve in dilute acids, but that the solution decomposes, after a short time, with the formation of a mixture of brown substances. An investigation of the action of moderately-concentrated hydrochloric acid on some indophenol paste, obtained from H. A. Schlieper, in Elberfeld, and prepared by Messrs. L. Casella and Company, in Frankfort-on-Maine, and which proved to be  $\alpha$ -naphthol blue, showed that the substance splits up into  $\alpha$ -naphthoquinone and dimethylparaphenylenediamine. Similarly the decomposition products of phenol blue were found to be benzoquinone and dimethylparaphenylenediamine. The reactions which take place may be expressed thus—



$\alpha$ -naphthol blue. The formation of  $\alpha$ -naphthol blue from dimethylparaphenylenediamine would then take place as below:—

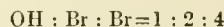


and similarly with phenol blue. In connection with this subject, the preparation of  $\alpha$ -naphthol blue from Biedermann's dibromo- $\alpha$ -naphthol and dimethylparaphenylenediamine, patented by L. Casella and Company, (111. Zusatz patent zu D. R.-P. 15915) is of considerable theoretical interest. The constitution of the dibromo- $\alpha$ -naphthol is probably

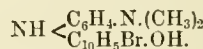


because (1) it yields phthalic acid on oxidation, so that the bromine atoms must be in the same benzene ring,

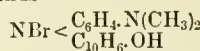
and (2) it is formed by direct bromination like dibromophenol, which has the structure



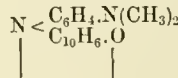
The formation of  $\alpha$ -naphthol blue probably takes place as follows: The bromine atom occupying the para position with regard to the hydroxyl unites with a hydrogen atom of the amido group in the molecule of the dimethylparaphenylenediamine, liberating hydrobromic acid and forming the compound



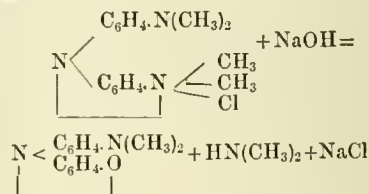
An alteration in position of the atoms may then be assumed to occur thus—



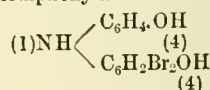
while lastly, hydrobromic acid is set free, and  $\alpha$ -naphthol blue



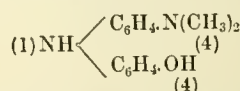
is formed. The relation between the substances resembling safranines and indophenols is shown by the action of caustic soda on Bindschelder's dimethylphenylene green, whereby phenol blue and diamethylamine are formed, thus—



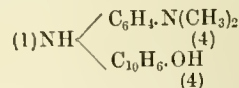
It appears probable that diphenylamine, diphenylhydro-sulfamine and phenyl- $\alpha$ -naphthalamine are related to the substances resembling indophenols, the indophenols, the group to which methylene blue belongs, the safranines, aniline black, and the indulines, in the same way that triphenylmethane and its homologues are related to the rosaniline, rosolic acid, and phthalein group. Thus leucodibromoquinonephenolimide may be considered as diparaoxydibromodiphenylamine—



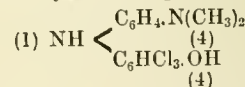
the leuco-compound of phenol blue or phenol white as dimethylparamidoparaoxydiphenylamine—



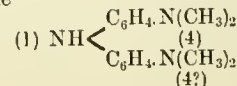
$\alpha$ -naphthol white as dimethylparamidoparaoxyphenyl- $\alpha$ -naphthylamine—



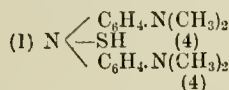
leucotrichloroquinonedimethylanilenimide as dimethylpara(?)amidoparaoxytrichlorodiphenylamine



leucodimethylphenylene green as tetramethyldiparamidodiphenylamine



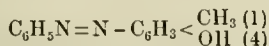
and leucomethylene blue or methylene white as tetramethyldiparamidodiphenylhydrosulphamine



The comparative facility with which diphenylamine and similar amido compounds may be prepared on the large scale makes it extremely probable that they will play an important part in the manufacture of colouring matters.—S. Y.

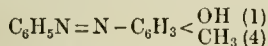
*On Azo and Disazo Compounds of the Cresols.* E. Nölting and O. Kohn. Berl. Ber. 17, 351.

WITH the exception of a paper by Mazzara, on phenylazoparacresol,

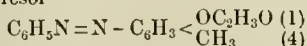


and a note by Stebbins, on phenylazocresolsulphonic acid, no work on the azo compounds of the cresols has been published. The experiments to be described show that paracresol combines with diazo compounds with as much ease as ortho- or metacresol. In the compounds formed the diazo group takes the ortho position with respect to the hydroxyl of the cresol. Neither diazo compounds nor nitroso compounds were obtained from paracresol. In the combination of ortho and metacresol with diazo compounds the azo group takes the para position with respect to the hydroxyl. Diazo compounds are readily formed from ortho and metacresol, and in these compounds, just as in the phenyldiazophenol prepared by Griess, the two azo groups occupy the ortho and para position with regard to the hydroxyl, and therefore the meta position with respect to each other. Nitroso compounds are also readily formed, the nitroso, like the azo group, taking the para position.

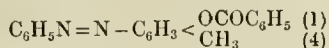
1. AZO COMPOUNDS OF PARACRESOL.—Phenylazoparacresol



The method of preparation described by Mazzara was modified, in so far that diazobenzene chloride was first prepared and poured into a concentrated alkaline solution of paracresol. Both liquids were cooled by the addition of ice. The quantities of the substances employed were: aniline, 20grms.; 30 per cent. hydrochloric acid, 52grms.; water, 400–450c.c.; titrated solution of sodium nitrate, equivalent to 15grms.  $\text{NaNO}_2$ ; paracresol, 22grms., dissolved in excess of caustic soda and diluted to 500c.c. An orange-yellow precipitate of phenylazoparacresol formed at once, and increased on standing. It was filtered after 24 hours, and after recrystallisation from benzene, or benzene and ligroin, was obtained in beautiful orange-yellow leaflets, melting, like Mazzara's product, at  $108^\circ$  to  $109^\circ$ . The composition was confirmed by analysis. Acids precipitate the substance unaltered from its alkaline solution. The yield is almost the theoretical. The substance dissolves with difficulty in concentrated, but easily in dilute warm alkalis. All attempts to prepare a disazo compound were fruitless. Phenylazopara-acetylcresol

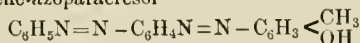


was prepared by heating phenylazoparacresol with slight excess of acetyl chloride or anhydride. The substance crystallises from dilute alcohol in fine yellow needles, melting at  $67^\circ$  to  $65^\circ$ . It is easily soluble in cold alcohol, ether, chloroform, and acetone. It is saponified slowly by carbonates, more rapidly by alkalis, but best by alcoholic hydrochloric acid, phenylazoparacresol being re-formed. The substance was analysed. The benzoyl compound

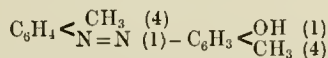


was prepared from benzoyl chloride dissolved in ether or benzene. It forms yellow needles, melting at  $113^\circ$ , easily soluble in boiling but with difficulty in cold alkalis; readily soluble in ether, chloroform, and hydro-

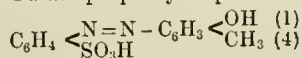
carbons. It is saponified with difficulty by alkalis. Azobenzene-azoparacresol



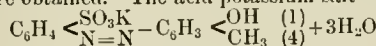
is not quite so easily prepared as the corresponding aniline compound. To a solution of amidoazobenzene hydrochloride in a little alcohol another molecule of hydrochloric acid was added, and the mixture poured into water, and treated, while cooled with ice and agitated, with sodium nitrite. After standing for some time the diazo compound was poured into a cooled alkaline solution of paracresol. After some hours the mixture was warmed. The azo compound was deposited, and was recrystallised from glacial acetic acid. It forms small brown needles, melting at  $160^\circ$ , soluble with difficulty in alcohol, more easily in chloroform and hydrocarbons, and readily in hot glacial acetic acid. It is not dissolved by alkalis, but forms a red-violet solution with sulphuric acid. It was analysed. Paratolylazoparacresol



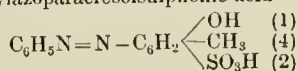
was prepared like the phenyl compound. It crystallises from toluene in reddish crystals or in yellow tablets, which melt at  $112^\circ$  to  $113^\circ$ . Difficultly soluble in cold, easily in hot alcohol, and in ether, chloroform, and hydrocarbons. The acetyl compound was obtained in yellow needles, melting at  $91^\circ$ , while the benzoyl compound melted at  $95^\circ$ . The same azocresol was prepared from Witt and Nölting's amidoazoparacresol, the constitution of which is known, hence the formula given is confirmed. Parasulphophenylazoparacresol



was formed by the action of diazobenzene sulphonic acid on an alkaline solution of paracresol. 8grms. of caustic soda, 32grms. of sulphanilic acid, and 12.1 grms. of sodium nitrite were dissolved in 200c.c. to 300c.c. of water, and to the cooled solution 43grms. to 45grms. of 30 per cent. hydrochloric acid were added. After standing a quarter of an hour the mixture was poured into a concentrated alkaline solution of 20grms. of paracresol. The acid sodium salt was precipitated from the dark yellowish-red solution by the addition of hydrochloric acid. After recrystallisation the free acid was prepared from the saturated aqueous solution by the addition of concentrated hydrochloric acid. It forms yellow-brown leaflets, which reflect violet light. It is easily soluble in cold water, moderately so in boiling alcohol, but almost insoluble in cold. It decomposes on heating. A number of well-crystallised salts were obtained. The acid potassium salt

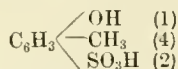


forms beautiful yellow leaflets, and is soluble in cold water. The corresponding sodium salt is very similar. The acid magnesium salt forms yellow-brown leaflets, which are insoluble in cold but dissolve easily in boiling water. The acid barium salt forms yellow-brown tabular crystals, insoluble in cold and but slightly soluble in hot water. The free acid and the acid solutions of its salts colour silk and wool a beautiful yellow, the tint being more orange than the corresponding phenol compounds. The same sulphonic acid was prepared by dissolving phenylazoparacresol in 4 to 5 times its weight of 100 per cent. sulphuric acid, and adding fuming sulphuric acid containing 60 to 65 per cent. of  $\text{SO}_3$ , in the proportion of one molecule  $\text{SO}_3$  to one of the cresol. The mixture was warmed until a drop of it gave a clear solution with water, when the free acid was precipitated by pouring into a small quantity of water. The properties of both the free acid and its salts were identical with those of the compounds just described, and their identity was further proved by the formation of sulphanilic acid by the reduction of the acid. Phenylazoparacresolsulphonic acid

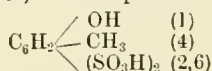




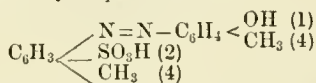
When paracresol is treated with ordinary sulphuric acid, the sulphonic acid



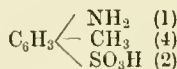
is formed. It unites readily with diazo compounds, forming colouring matters soluble in water. The above compound was formed by its union with diazobenzene-chloride. It was obtained after recrystallisation in small red-brown tablets or needles, easily soluble in cold water, but with difficulty in alcohol. The acid sodium salt forms red-brown scales, readily soluble in hot and moderately in cold water. The acid solution imparts to wool and silk the same colour as the isomeric compound from sulphanic acid. Neither diazobenzene chloride nor diazobenzene sulphonic acid combines with the paraeresoldisulphonic acid prepared by Engelhardt and Latschinoff, which the authors, by converting it into the corresponding dinitro compound, found to possess the constitution



Metasulphoparatolylazoparacresol

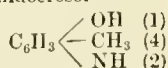


was prepared from the diazo compound of paratoluidine-sulphonic acid



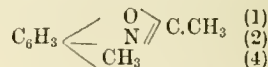
in the same way as the compound from sulphanilic acid. It forms red-brown crystals, which reflect violet light strongly. It dissolves easily in water, but with difficulty in alcohol. The acid sodium and barium salts were prepared. The sodium salt forms yellow scales soluble in cold water. The barium salt crystallises from dilute hydrochloric acid in small red-brown needles, almost insoluble in cold and but slightly in hot water.

**Reduction of Azoparacresol Compounds:** This was carried out with tin and hydrochloric acid, or with an acid solution of stannous chloride. The products obtained from phenylazoparacresol were aniline and amidoparacresol. Though the separation of these products was not easy, yet the quantity of the amidoazocresol was sufficient to establish its identity with that obtained from the sulphanilic acid. Since the same amidoeresol was always obtained, the authors conclude that only a single azo compound is formed by the union of diazo compounds with paracresol. The free acid or the barium salts was warmed on the water-bath with tin and hydrochloric acid, or with an acid solution of stannous chloride, until the yellow colour had disappeared. After evaporation to a small bulk, the sulphanilic acid or barium salt was filtered, the filtrate diluted with water, the tin precipitated with sulphuretted hydrogen, and the liquid evaporated in a current of sulphuretted hydrogen. The amidoeresol was then precipitated from the concentrated solution with sodium carbonate. The mother-liquor contained small quantities of amidoeresol, which was extracted with ether. The amidoeresol was rapidly filtered, dried in vacuo over sulphuric acid, dissolved in absolute ether, and precipitated with hydrochloric acid. The chloride was filtered, drained, and washed with absolute ether. It was thus obtained as a white powder, which was quite stable even in the air. The operations must be conducted quickly, for the amidoeresol and the hydrochloride, when impure or in solution, readily form tarry compounds. The chloride is easily soluble in water and alcohol, but insoluble in absolute ether and benzene. It begins to sublime on the water bath. Ferric chloride colours its aqueous solution red, but gives no such reaction with free amidoeresol. The substance was analysed. Amidoeresol

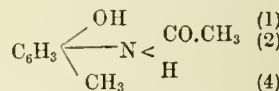


was precipitated from the chloride with sodium bi-

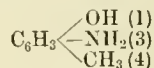
carbonate, and crystallised from ether or benzene. It forms glittering white scales, melting at 135°, which are stable in the air. It is easily soluble in alcohol, ether, and chloroform, but much less so in hydrocarbons and in water. It sublimes in white scales or needles, which melt at 135°. The composition was confirmed by analysis, and its constitution was proved by a comparison of its properties with those of the two possible amidoeresols. It was found to be identical with the ortho-amido compound ( $\text{OH} : \text{NH}_2 = 1 : 2$ ) investigated by Wagner, Hofmann, and Miller. The same compound was prepared by the authors from the nitroparacresol, melting at 34°. The methenyl and ethenyl compounds were prepared. Ethenylamidoparacresol



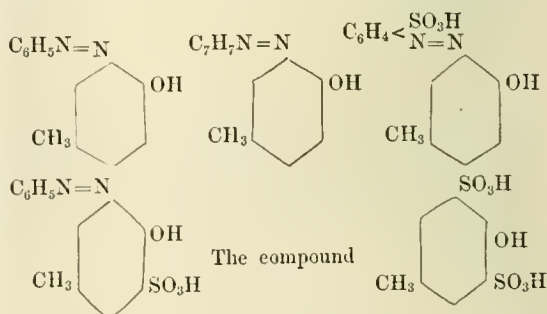
was obtained by heating amidoparacresol-chloride with excess of acetic anhydride and anhydrous sodium acetate for several hours in a flask with a reversed condenser. The compound boiled after repeated distillation at 218°–219°. It possesses a characteristic odour, and it is difficultly soluble in water, but easily in alcohol and ether. It is soluble in acids, but the salts are unstable and liberate the free base. On heating they yield acetyl-amidoeresol. The chloride and chloroplatinate were prepared and analysed. Acetylamidoparacresol



is easily obtained by warming a solution of the ethenyl base in dilute sulphuric acid, in the form of long white needles. After recrystallisation from water the substance melted at 159°–160°. It is very slightly soluble in water, alcohol, benzene, and ether in the cold, but more so on heating. The substance was analysed. The constitution of the amidoeresol is thus confirmed. The isomeric compound



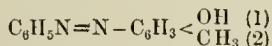
melts at 143°–144°. **Constitution of Azoparacresol Compounds:** It follows from the experiments described that in the azo compounds of paracresol the azo group occupies the ortho position with regard to the hydroxyl, just as in the derivatives of  $\beta$ -naphthol described by Liebermann and Jacobsen (*Berl. Ber.* 14, 1310 and 1791). The authors also succeeded in preparing paratolylazoparacresol from amidoazoparatoluol, but the yield was very small. The amido group in amidoazo compounds seems thus to be much less easily replaced by hydroxyl than in the amines, for with the latter very good results are obtained. The formation of paratolylazoparacresol is a further proof of the constitution of the azoparacresol compounds. The structure of these compounds is thus represented—



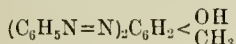
does not combine with diazo compounds.

2. AZO COMPOUNDS OF ORTHOCRESOL.—When diazobenzene chloride was added to an alkaline solution of

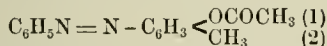
orthoeresol, only a small precipitate was formed. The phenylazo-orthoeresol



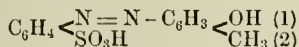
was precipitated with an acid and recrystallised from a mixture of benzene and ligroin or dilute alcohol. It forms beautiful yellow glittering scales, melting at 129°–130°, insoluble in cold, difficultly in boiling water, easily soluble in alcohol, ether, chloroform and hydrocarbons, but less so in ligroin. Like phenylazophenol it has still feeble acid properties: it does not decompose carbonates, but dissolves in cold dilute alkalis and ammonia with a yellow-red colour. The lead salt was prepared. The composition was proved by analysis. The product precipitated in small quantity directly from the alkaline solution consisted of a mixture of the sodium salt of phenylazo-orthoeresol and phenyldisazo-orthoeresol. It was treated with dilute acid, and then with cold alcohol, in which the azo compound dissolves readily, while the disazo compound is almost insoluble. By recrystallisation from hot alcohol phenyldisazo-orthoeresol



was obtained in red-brown scales, melting at 114°–115°, difficultly soluble in cold, more easily in boiling alcohol, but generally less soluble than the azo compound. With warm alkalis it forms a yellowish-red solution, but it is insoluble in carbonates and in ammonia. It forms a red solution with cold sulphuric acid. The same substance is more easily prepared by the action of two molecules of diazobenzene chloride on an alkaline solution of orthoeresol. The greater part separates at once—the rest is precipitated by the addition of an acid. The azo compound is frequently formed as well as the disazo compound. The composition was confirmed by analysis. An acetyl compound may be prepared in the usual way: it crystallises from dilute alcohol in beautiful yellow needles, which melt at 120°–121°, and dissolve easily in cold alcohol, ether, and benzene. When heated with alkalis or hydrochloric acid the original substance is re-formed. Phenylazo-orthoacetyleresol

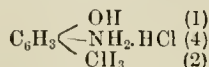


is obtained by heating the azo compound with acetic anhydride or chloride. It crystallises from dilute alcohol in beautiful yellow tablets, melting at 81°–82°, and moderately soluble in cold alcohol, ether, chloroform, and aromatic hydrocarbons. It is saponified with difficulty by carbonates, more easily by dilute alkalis or alcoholic hydrochloric acid. The substance was analysed. The corresponding benzoyl compound forms small yellow needles, which melt at 110°–111°, and dissolve with difficulty in cold but readily in boiling alcohol, also in ether, chloroform, and acetone. It is not saponified by carbonates, and very slowly by alkalis. It was analysed. Parasulphophenylazoorthoeresol



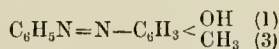
was prepared like the corresponding compound from paracresol. The free acid crystallises from very dilute hydrochloric acid in small red-brown needles, soluble with difficulty in cold, easily in hot water, and almost insoluble in alcohol. The acid sodium and barium salts were prepared and analysed. *Reduction of Azo-orthoeresol Compounds:* It was to be expected that in the reduction of these compounds the amido group in the amidocresol would occupy the para position with regard to the hydroxyl, and this was found to be the case. The method of reduction was the same as in the previous case. The amidocresol crystallised from benzene in small white scales, which melted at 172°–173°. After sublimation the melting point was 174°–175°. It is easily soluble in alcohol and ether, less so in water and hydrocarbons. By its oxidation with chromic acid toluquinone, melting at 67°–68°, was formed, and the constitution was thus proved. The same amidocresol was

obtained by the authors by reduction of nitroso-orthoeresol, in which the nitroso group was known to occupy the para position. By treating an ethereal solution of the base with gaseous hydrochloric acid, amido-orthoeresol hydrochloride

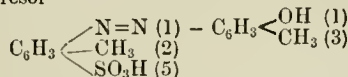


was precipitated as a white crystalline powder, easily soluble in water and alcohol. It is not coloured by ferric chloride, but on warming the solution quinone is formed. The hydrochloride was analysed.

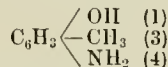
3. AZO COMPOUNDS OF METACRESOL.—Phenylazo-metacresol



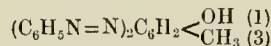
By the action of diazobenzene chloride on an alkaline solution of metacresol, an insoluble precipitate and a highly-coloured solution were obtained. The precipitate consisted chiefly of the disazo compound, while the azo compound was contained in the solution, and was separated and purified in the usual way. It crystallises from ligroin in beautiful yellow needles, melting at 109°. It is easily soluble in alcohol, ether, chloroform, and hydrocarbons. It forms yellowish-red salts with alkalis and ammonia, but does not decompose carbonates. The composition was confirmed by analysis. Sulpho-orthotolyl-azometacresol—



was obtained in the usual way from metacresol and the sulphonic acid of orthotoluidine prepared by Neville and Winther. The free acid forms small red-brown crystals, which reflect violet light. It dissolves readily in cold water, less so in cold but easily in hot alcohol. The acid sodium and barium salts were prepared and analysed. The former is easily soluble in water, the latter almost insoluble in cold but moderately soluble in boiling water. The azo compounds of ortho- and meta-cresol colour silk and wool an orange-yellow. *Reduction of Azometacresol Compounds:* An amidometacresol was obtained in the usual way. It melted at 151°, and yielded a toluquinone melting at 67° to 68°. Its constitution must therefore be—



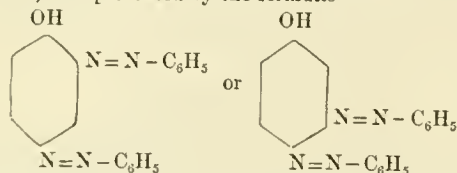
and the azo group therefore occupies the para position with respect to the hydroxyl in the azo compounds of metacresol. Phenyldisazometacresol



The insoluble precipitate mentioned above was purified in the same manner as the corresponding compound from orthoeresol. The same compound was prepared by adding two molecules of diazobenzene to an alkaline solution of metacresol. It crystallises from alcohol in red-brown scales, melting at 149°. In this case, again, the melting point of the disazo is higher than that of the azo compound. It is difficultly soluble in cold, more easily in boiling alcohol; soluble in ether, chloroform, and benzene. It dissolves in warm dilute caustic potash. The composition was proved by analysis. The acetyl compound was also prepared. It crystallises from dilute alcohol in small yellow-brown needles, melting at 156° to 157°. *Constitution of the Disazo Compounds of the Phenols and Cresols:* The reduction of these compounds to the corresponding diamines offers the best means of determining the position of the second azo group. The diamido phenols are very unstable, but their ethers are less easily decomposed. Both the alcoholic and acid ethers of the amidophenols possess the properties of amines. As the one azo group, both in the phenol and in the two cresol derivatives, occupies the para position with regard to the



hydroxyl, the second must take either the ortho or meta position, as represented by the formulæ—



and the corresponding diamines must behave as meta or orthodiamines. The first of these formulæ has been experimentally established. Phenyl-disazomethylphenol,  $(\text{C}_6\text{H}_5\text{N}=\text{N})_2\text{C}_6\text{H}_3\text{OCH}_3$ , was prepared by dissolving 3 grms. of phenyl-disazophenol in 20 c.c. of absolute alcohol, adding 0.4 grms. of potassium, and, after cessation of the evolution of hydrogen 2 grms. to 2.5 grms. of methyl iodide. After heating for some hours on the water bath, the alcohol was removed by evaporation, the residue freed from potassium iodide by washing with water, and the purified product recrystallised from ligroin, to which a little animal charcoal was added. The substance dissolved with difficulty in cold but easily in boiling alcohol, and in acetone, chloroform, ether, and benzene. It melts at  $110^\circ$ . Phenyl-disazophenol is reproduced by warming with alkalis. The substance was analysed. The acetyl compound, prepared in the ordinary way, crystallised from alcohol in small yellow needles, melting at  $116^\circ$ , and soluble in alcohol, ether, chloroform, and benzene. It is easily saponified by alkalis or alcoholic hydrochloric acid, with difficulty by carbonates. The benzoyl compound was prepared from benzoyl chloride. It crystallises from alcohol in small yellow needles, which melt at  $138^\circ$  to  $139^\circ$ . It is not easily saponified by alkalis. *Reduction of Disazo Compounds:* The ethers of the disazo compounds were dissolved in cold glacial acetic acid, and reduced in the cold with zinc dust. The solutions were diluted with water, filtered from zinc dust, and tested directly. The presence of the primary bases is not prejudicial to the reactions of the meta, or for the most part of the para diamines. A brown coloration was produced by the addition of a nitrite to the solution acidified with a few drops of hydrochloric acid. Diazobenzene chloride produced an orange-yellow coloration, and nitrosodimethylaniline, or better dimethylphenylenediamine, and an oxidising agent gave Witt's neutral tint reaction. The two amido groups therefore occupied the meta position with regard to each other. The three ethers of phenyl-disazophenol and the acetyl derivatives of both diazoacresols gave precisely the same reactions—hence the position of the amido groups is the same in all. Since para-cresol forms no disazo compound, it must be concluded that one but not two azo groups can occupy the ortho position with regard to the hydroxyl.—S. Y.

*On the Oxidation of Purpurine.* Chr. Dralle. Berl. Ber. 17, 376.

It is well known that the purpurine obtained from madder is less stable than the isomeric compounds prepared synthetically, and far less so than alizarin. Schunk and Römer observed that a solution of purpurine in caustic potash lost its colour when exposed to the light. It was not known what product was found, but the author, repeating the experiment, recognised the presence of phthalic acid in the solution. Phthalic acid is also formed by the oxidation of an alkaline solution of purpurine by means of potassium ferricyanide. Quinizarin, under the same conditions, yields phthalic acid, but alizarin gives no crystalline products which can be extracted with ether.—S. Y.

*Note on Chrysaniline.* R. Anschütz. Berl. Ber. 17, 433.

(The author describes experiments on chrysaniline made in conjunction with Dr. Oscar Müller.)

In order to ascertain the number of amido groups in chrysaniline the acetyl compound was prepared by heating one part of crude chrysaniline with  $2\frac{1}{2}$  parts of acetic anhydride, in sealed tubes, from  $140^\circ$  to  $160^\circ$  for 8 to 12 hours. The product, which still possessed basic proper-

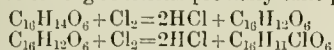
ties, was poured into water, and the resinous substance boiled repeatedly with water and filtered while hot. The hydrochloride was precipitated from the filtrate by the addition of hydrochloric acid, and the acetyl compound liberated by means of caustic soda. It was almost insoluble in water, but dissolved in alcohol. From the alcoholic solution, which exhibited a blue fluorescence, the substance was precipitated in the form of microscopic needles by careful addition of water. The analysis of the compound agreed with the formula  $\text{C}_{19}\text{H}_{13}\text{N}_3(\text{C}_2\text{H}_3\text{O})_2$ , so that there are probably two amido groups present in the molecule. The hydrochloride and nitrate of the acetyl compound were prepared and analysed. The former occurs in yellow flakes, consisting of minute long yellow needles. It is easily soluble in hot water, much more so than in dilute hydrochloric acid, by which it is saponified on heating. It dyes silk and wool yellow. The nitrate was prepared by the addition of silver nitrate to the hydrochloride. Pure chrysaniline was prepared by boiling one part of the pure acetyl compound with three parts of hydrochloric acid (of specific gravity 1.12) and six parts of water for eight hours. The residue after evaporation was dissolved in water and precipitated with nitric acid. The hot aqueous solution of the nitrate was treated with caustic soda, and the precipitated chrysaniline recrystallised from dilute alcohol, when it was obtained in prismatic needles. The analysis of the compound agreed fairly well with the formula  $\text{C}_{19}\text{H}_{13}\text{N}_3$ . The properties of chrysaniline are very little altered by its conversion into the acetyl compound. The latter possesses powerful basic properties, and forms salts which are very similar to those of chrysaniline. The hydrochloride, for instance, is a colouring matter. The author and Dr. Müller considered that the basic properties of the acetyl compound were due to the similarity in position of the third nitrogen atom to that in the molecule of pyridine or quinoline. The effect of oxidising agents on chrysaniline was therefore studied. Potassium permanganate yielded only oxalic acid, but potassium bichromate and sulphuric acid gave better results. One part of chrysaniline was dissolved in nine parts of sulphuric acid diluted with fourteen parts of water, and to the solution six parts of bichromate were gradually added. The reaction was completed by boiling the mixture a few minutes. The precipitate was filtered, dissolved in an alkali, and treated while cold with potassium permanganate so long as the red colour changed to green. After removal of excess of permanganate by boiling with alcohol the solution was filtered and precipitated with hydrochloric acid. The precipitate was dried, mixed with twenty parts of lime, and distilled. Acridine and at least two other bases were found in the distillate, but the quantity of only one of them was large enough for investigation. The hydrochloric acid solution of the distillation products was treated with ammonia, when the bases separated in the liquid state, but after standing a few days, white leafy crystals were deposited, together with brown masses. The crystals were purified by sublimation, and were found to be difficultly soluble in water, but easily in alcohol. They melted at  $148^\circ$ – $150^\circ$ . The picrate was much more easily soluble in alcohol than acridine picrate. The nature of the other substances was not ascertained, but it was thought possible that an amido-acridine might be formed by the distillation with lime of the oxidation products of chrysaniline. Amidoacridine was therefore prepared by the reduction of the nitroacridine, melting at  $215^\circ$ , with tin and hydrochloric acid. It melted at  $209^\circ$ . The picrate was a scarlet substance, soluble in cold alcohol, and crystallising from hot alcohol in garnet-red prisms, which, when examined under the microscope, were found to resemble the picrate formed from the brown masses mentioned above.

S. Y.

*On Haematoxylin and Brasilin.* Chr. Dralle. Berl. Ber. 17, 372.

THE action of various reagents on haematoxylin and brasilin was studied with the view to obtain products, which might throw a light on the structure of these compounds. The results, however, were not very satis-

factory. (1) *Haematoxylin*: By heating with ten parts of concentrated hydrochloric acid, in sealed tubes, to 110° to 115°, and by the action of 40 per cent. hydrobromic acid, colourless crystals were obtained embedded in a black smeary mass, from which they could not be separated by means of any solvent. The action of chlorine gas on an aqueous solution of haematoxylin yielded no definite product. By the action of titrated chlorine water on the aqueous solution various chlorine substitution products were obtained, but they could not be crystallised. With two molecules of chlorine to one of haematoxylin a product was obtained which contained in one case 9.09 and in another 10.26 per cent. of chlorine, while monochlorohaematein requires 10.61 per cent. The following reactions probably take place:—



At the same time one molecule of chlorine forms chlorine products, while according to the above reactions only haematein should be formed. The author does not consider the product analysed to be pure monochlorohaematein. The product obtained by the action of dry chlorine on dry haematoxylin contained only 6 per cent. of chlorine, but by the action of chlorine water on the aqueous solution a product containing 17.78 of chlorine was formed. It was not, however, a definite compound. By the addition of four molecules of bromine dissolved in glacial acetic acid to a hot solution of one molecule of haematoxylin in the same substance, the solution became cherry red, and soon deposited deep-red pointed crystals, which were washed with glacial acetic acid, and then with ether, and dried quickly at 100°. They decomposed at 120°, and could not be recrystallised. The analysis agreed with the formula  $\text{C}_{16}\text{H}_{12}\text{Br}_4\text{O}_6$ . The bromine compound dissolves in dilute alkalis and in water with a brown-red colour and without decomposition. Haematoxylin is not reformed by neutralisation with sulphuric acid, hence the bromine compound is a substitution and not an addition product. No acetyl compound could be obtained from the dibromo compound; and by fusion with potash only formic and oxalic acid were formed, besides tarry and carbonaceous matters. Nitric acid gave no result. Concentrated, or better fuming, sulphuric acid gave a sulphonic acid, which was not further investigated. The action of zinc dust was found to be similar to that described by Reim, except that the evolution of carbon dioxide and of a gas burning with a blue flame was observed, as was also the presence of resorcin in the distillate. Phosphorus, hydriodic acid, methyl iodide in presence of methyl alcohol and two molecules of caustic potash, as well as ethyl iodide under the same conditions, gave no result. Diazobenzene hydrochloride added to a solution of one molecule of haematoxylin in two of caustic potash gave a yellow-brown precipitate, which contained nitrogen, and was probably haematoxylindiazobenzene, but it could not be crystallised. Other diazo compounds might, perhaps, give better results. Acetyl haematoxylin was then examined. When treated with potassium permanganate no acetyl haematein was formed, but only carbon dioxide and oxalic acid. Nitric acid and chlorine gave no results. By heating one part of acetyl haematoxylin with 3.03 parts of a 20 per cent. solution of bromine in glacial acetic acid to 100° to 110°, in sealed tubes, for 3 hours to 4 hours, crystals were obtained, which, when washed with glacial acetic acid and ether, and dried over caustic soda and then calcium chloride, gave numbers on analysis which agreed with the formula  $\text{C}_{16}\text{H}_8\text{Br}_4\text{O}_6(\text{C}_2\text{H}_3\text{O})_5$ . The yield was, however, only 15 per cent. to 20 per cent. of the theoretical amount. With 4 molecules of bromine no crystals were obtained. The product decomposes at 118° without melting. *Brasilin*: After treatment with hydrochloric acid long needles were deposited from the acetic acid solution, but they could not be recrystallised. Hydrobromic acid and sulphuric acid gave the same results as with haematoxylin. Chlorine and bromine gave no definite products. By fusion with potash, oxalic acid, formic acid, and considerable quantities of resorcin were formed. Resorcin was also formed by heating brasilin with zinc dust. It is a product of the simple distillation of brasilin (Kopp),

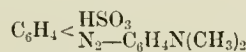
*Berl. Ber.* 4, 334). With methyl iodide and caustic potash—4 molecules of alkali to 1 molecule of brasilin—a brick-red compound was obtained, the spectrum of which differed widely from that of brasilin, while the reactions with ferric chloride and with alkalis were no longer observed. The alcoholic solution was fluorescent. The new substance was doubtless a methylbrasilin, but it could not be obtained sufficiently pure for analysis. Diazobenzenehydrochloride yielded a small quantity of brasilnazobenzene, which, however, could not be purified. Attempts were made to obtain acetylbrasilein from acetylbrasilin by the action of potassium manganate, nitric acid, and bromine, and by that of acetylchloride on brasilein, but without result.—S. Y.

*Improvements in Colouring Matters.* Frank Wirth, Frankfort. A communication from Messrs. Dittler and Company, Farbwerk Griesheim-am-Main, near Frankfort, Germany. Eng. Pat. 2104, April 25, 1883.

NAPHTHALENE is heated with five times its weight of concentrated sulphuric acid to a temperature of 160° C. for four hours. The mixture of naphthalene-disulphonic acids thus obtained is nitrated when cool by the addition of nine parts of nitric acid and the nitrosulphonic acids diluted and reduced, timed, and converted into sodium salts. Other methods of sulphonating, by using a smaller quantity of fuming sulphuric acid, are also mentioned. The amidosulphonic acids thus obtained are diazotised in the usual manner and combined with phenol or the naphthols, and thus produce yellow or red dyes.—R. M.

*A New or Improved Process for the Manufacture of Blue Colouring Matters Suitable for Dyeing and Printing.* Herbert John Hadden. A communication from Lembach and Schleicher, Biebrich-on-the-Rhine, Germany. Eng. Pat. 3491, July 16, 1883. Provisional protection only.

PARA-AZOBENZENE SULPHONIC ACID is combined with dimethyl-aniline so as to produce the well-known azo-sulphonic acid



The latter is dissolved in ammonia, reduced with zinc dust, filtered, the solution acidulated, and then saturated with  $\text{H}_2\text{S}$ . Oxidation is then effected by means of ferric chloride, and the colouring matter precipitated by zinc chloride.—R. M.

*Improvements in Colouring Matters.* Frank Wirth. A communication from Heinrich Baum, Höchst-on-the-Main, Germany. Eng. Pat. 3498, July 16, 1883. Provisional protection only.

WHEN alphanaphthol is treated with from one to two parts of concentrated or fuming sulphuric acid "at any temperature, and in any manner, a mixture is almost always obtained of sulpho acids, which mixture, together with diazo bodies, yields only a small percentage of useful azo colours." The reason of this, according to the inventor, is to be found in the circumstance that "alphanaphthol, as compared with the betanaphthol, forms a very constant, and difficultly soluble alphanaphthol sulphonic acid ( $\alpha\text{-C}_{10}\text{H}_7\text{OH}\cdot\text{SO}_3\text{H}$ ), which in the presence of hydrated sulphuric acid becomes sulphurised (? sulphonated), and then by further heating yields an alphanaphtholdisulpho acid, which is useless for the production of azo colours." To obviate this the inventor first converts the naphthol into its acetyl derivative by heating with acetic anhydride and then adds the theoretical quantity of sulphuric acid (or "mono-chlorhydine") to form a monosulphonic acid. Two isomeric monosulphonic acids are said to be produced by this means, the one giving a soluble and the other an almost insoluble barium salt. The acid giving an easily soluble barium salt, furnishes azo dyes with the diazo compounds of the aromatic hydrocarbons. The difficultly soluble acid, on the other hand, is stated to give no useful dyes with the diazo compounds of the aromatic hydrocarbons, but with



their diazo sulphonie and carbonic acids it yields valuable crystalline products. Detailed examples are given in the specification, and the inventor adds a long list of the various azo colours produced by the two isomeric monosulphonie acids.—R.M.

*Improvements in Colouring Matter.* Frank Wirth. A communication from Heinrich Baum, Höchst-on-the-Main, Germany. Eng. Pat. 3523, July 17, 1883. Provisional protection only.

THE inventor claims the production of a betanaphthol-disulphonie acid by the following process: The potassium salt of Schätfler's betanaphtholmonosulphonie acid is mixed intimately with an equal weight of potassium pyrosulphate, and the mixture heated with strong sulphuric acid to 120° to 130° C. until completely dissolved. The temperature is then raised and maintained at 150° to 160° C. for five or six hours. Only one disulphonie acid is said to be produced, and the inventor employs this for the manufacture of azo colours in the usual way. A list of the colours produced by combination with various diazo compounds is given in the specification.—R.M.

*Improvements in Colouring Matter.* Frank Wirth. A communication from Heinrich Baum, Höchst-on-the-Main, Germany. Eng. Pat. 3527, July 17, 1883. Provisional protection only.

THESE colouring matters are produced by treating the dry haloid salts of primary, secondary, and tertiary aromatic monamines with organic acid anhydrides, the novelty of the invention being the occurrence of the reaction without the use of dehydrating agents such as  $\text{ZnCl}_2$ , etc. Thus, in the case of aniline, the dry hydrochloride is heated with  $1\frac{1}{2}$  to twice its weight of acetic anhydride, in a closed vessel, to 180° to 200° C. for twelve hours. The resinous melt is boiled with dilute sulphuric acid, allowed to cool, filtered, and the base thrown out by alkali. The hydrochloride of the base is prepared for commerce, and is a greenish-yellow dyestuff.—R.M.

*Improvements in the Manufacture of certain Sulphonie Acids.* Frank Wirth. A communication from the Colour Factory (formerly Brönner) of Frankfurt-on-the-Main, Germany. Eng. Pat. 3606, July 23, 1883.

THE object of the invention is the preparation of sulphonie acids of betanaphthol from the betadinaphthyl ether,  $\text{C}_{20}\text{H}_{14}\text{O}$ , which is obtained in the manufacture of betanaphthol. The conversion is effected by heating the ether with ordinary sulphuric acid, or with sulphuric acid containing 10 per cent. of anhydride, according as the mono- or di-sulphonie acid is required.—R.M.

*Improvements in the Production of Certain Colouring Matters applicable to Dyeing and Printing.* Thomas Holliday, Huddersfield. Eng. Pat. 3971, August 16, 1883.

THIS invention claims the employment of the fluorols and their sulphonie acids for the production of azo colours. The fluorols are combined with the diazo salts of benzene, toluene, xylene, etc., in precisely the same way as in the case of the naphthols. By the employment of fluorylamine a series of azo colours analogous to the naphthylamine compounds is said to be obtained.—R.M.

*Production of a New Solid Base by Reaction of Aldehyde and Hydrochlorate of Aniline, or their equivalents.* John Imray. A communication from the Actien Gesellschaft für Anilin-Fabrikation, Berlin. Eng. Pat. 4207, Aug. 31, 1883.

EIGHT kilos. of aniline hydrochloride are dissolved in 16 litres of water and mixed with a cold dilute solution of aldehyde. The reaction is complete in three or four days, and on evaporating the solution the hydrochloride of the new base,  $\text{C}_{18}\text{H}_{20}\text{N}_2$ , is obtained in a crude state, and is purified by precipitation with alkali. This base, when the dry hydrochloride is treated alone or in the presence of metallic chlorides, is converted into the hydrochloride

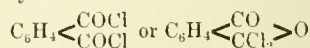
of quinaldine. Instead of aldehyde, paraldehyde, aldol, acetal- or croton- aldehyde can be used, and the aniline may be replaced by other aromatic bases. In the ordinary preparation of quinaldine, aldehyde is made to act upon a hot solution of aniline in the presence of hydrochloric acid.—R. M.

*An Improvement in the Manufacture of Sulphoconjugate Paris Violet Dyes.* John Imray. A communication from the Société Anonyme des Matières Colorantes et Produits Chimiques de Saint Denis. Messrs. A. Poirrier and A. Rosenstiehl, Paris. Eng. Pat. 903, Jan. 7, 1884.

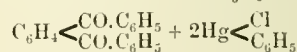
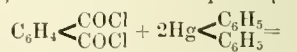
IN order to avoid the large excess of sulphuric acid which has hitherto been employed in preparing the sulpho acid of the Paris violets the inventors produce "from the sulphuric acid in whole or in part soluble sulphates" such as those of potassium, sodium, magnesium, zinc, etc., "no more water being added than is necessary to form a paste of suitable consistence. By this treatment there is no alteration of the colouring matter, and it is obtained in a state in which, by reason of its great solubility, it can be employed with great facility."—R.M.

I. *On Orthonitrobenzyl Chloride.* Communications by E. Nölting, Berl. Ber. 17, 385.

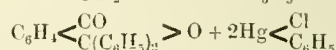
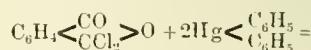
BEILSTEIN and Geitner observed that in the nitration of benzyl chloride, in addition to crystallised paranitrobenzyl chloride, an oil was formed, which appeared, from its analysis, to be an isomeric compound. Having some quantity of this oil at his disposal, the author subjected it to oxidation by means of potassium permanganate, and obtained, besides some benzoic acid and paranitrobenzoic acid, a large quantity of orthonitrobenzoic acid. After removal of benzoic acid with steam, the separation of the ortho and para compounds was easy on account of their different solubility, as well as that of their barium salts. The presence of metanitrobenzoic acid was not observed. The ortho compound was recognised by its melting point, and by the properties and analysis of its barium salt. At 15°, according to Strakosch, the product is almost entirely paranitrobenzyl chloride. At ordinary temperatures a considerable quantity of the ortho compound is formed. Since the nitrobenzyl chlorides are much more easily oxidised by permanganate than the nitrotoluenes, they form a convenient starting point in the preparation of para- and ortho- nitrobenzoic acid. Some attempts to prepare orthonitrobenzaldehyde from the oil were unsuccessful. [For communication II, see under Destructive Distillation, etc.] III. *On the Constitution of Phthalyl Chloride* (by the author and G. von Bechi): The constitution of phthalyl chloride must be represented by one of the two formulæ—



The known constitution of phthalophenone and phthalide has generally induced chemists to adopt the latter formula, but no direct proof has been adduced unless the difference observed by Graebe between the ethers obtained from phthalates and from phthalyl chloride be taken as such. The authors attempted to solve the problem by treating phthalyl chloride with mercury diphenyl. The formation of the diketone, or of phthalophenone, as represented below, should afford the required proof—



or

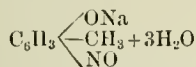


Unfortunately either large quantities of the original substances remained unacted on or complete decomposition took place. Phthalophenone was, however, obtained in small quantity, while no substance having the properties of an aromatic diketone was produced. The following

method gave the best results: 6grms. of phthalyl chloride were heated with 20grms. of mercury diphenyl, and 20 to 30grms. of benzene in closed tubes to 150° to 160° for twelve hours. After cooling, the crystals of mercury phenylchloride were filtered off and washed with benzene, and the latter was evaporated. The residue was boiled with concentrated hydrochloric acid to destroy the mercury compounds, and to convert the unaltered phthalyl chloride into phthalic acid. The residue was boiled first with water, then with soda, and the small residue recrystallised from alcohol in presence of animal charcoal. Pure phthalophenone was thus obtained. It melted at 112°, and a comparative microcrystallographic examination by Prof. Lehmann of the product, with that obtained by the method of Friedel and Crafts, established the identity of the substances.—S. Y.

*On Nitroso-orthocresol.* E. Nölting and O. Kohn, Berl. Ber. 17, 370.

NITROSO-ORTHO-CRESOL is easily obtained by the method of Stenhouse and Groves (Ann. Chem. Pharm. 188, 360). The orthocresol was dissolved in from 30 to 40 times its quantity of water, and treated, drop by drop, in the cold with the theoretical quantity of a titrated solution of nitrosyl sulphate, the mixture being constantly stirred. After a time a gray-white precipitate of the nitroso compound was formed, while a small quantity of tarry matter collected on the surface of the liquid and was easily removed. The filtered nitrosocresol was dissolved in ammonia, purified with carbon dioxide, and poured into ice-cold dilute sulphuric acid. The substance recrystallises from benzene in light gray warty masses, and from water in long white needles. It dissolves with difficulty in cold but easily in boiling water, and in alcohol, ether, and chloroform. It is less soluble in benzene. With dilute alkalis it forms a red-brown solution, from which it is precipitated by acids as a white flocculent precipitate. It melts at 134° to 135°, being at the same time decomposed. It was analysed. The sodium salt



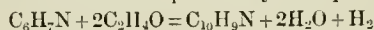
obtained by the action of sodium on the ethereal solution of the nitroso compound crystallises from acetone in brown needles. It dissolves easily in water and alcohol, forming red-brown needles. It deflagrates on heating, but the water of crystallisation may be removed in a desiccator containing sulphuric acid. The potassium and silver salts were also prepared. The salts of the heavy metals give with the alkali salts of the nitroso compound amorphous precipitates of various colours. By its oxidation with potassium ferricyanide, nitroso-orthocresol yielded the nitro-orthocresol melting at 94° to 95°, while with nitric acid, of specific gravity 1.33°, the dinitro-orthocresol melting at 85° to 86° was formed. By its reduction the amidocresol, melting at 174° to 175°, was formed. The same compound was also formed by the reduction of the azo compounds of orthocresol. The amido cresol yielded toluylquinone on oxidation. It is thus proved, both by its oxidation and reduction, that in the nitroso-orthocresol the nitroso group occupies the para position with regard to the hydroxyl.—S. Y.

*On the Azo Colouring Matters.* G. Schultz, Berl. Ber. 17, 461.

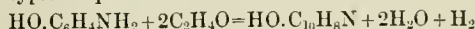
ACCORDING to S. Pfaff, the so-called alpha-monosulphonic acid from beta-naphthol differs from the isomeric compound obtained by Schäffer, inasmuch as it does not unite with diazoxylene under ordinary conditions, i.e., in dilute solution to form an azo colouring matter, while it does unite with other diazo compounds—for instance, with diazobenzene sulphonic acid. The author finds, however, that when a concentrated solution is employed, xyleneazobeta-naphthol sulphonic acid is obtained in the form of red needles, which, like the isomeric compound, dissolve in concentrated sulphuric acid with a cherry-red colour. The colour imparted to wool is somewhat yellower than that produced by the

isomeric colouring matter from Schäffer's acid. The behaviour of the tetrazodiphenyl obtained from benzene towards the betanaphthol disulphonic acid, which forms a sodium salt insoluble in alcohol (the so-called disulphonic acid R), is very remarkable. When one molecule of a salt of tetrazodiphenyl is added to an alkaline solution of one molecule of the above-mentioned disulphonic acid, a red colouring matter is obtained, which is completely precipitated by common salt. Neither of the original substances can be detected in the filtrate. A blue substance is not formed by warming the colouring matter. With two molecules of a cold dilute alkaline solution of the betanaphthol sulphonic acid to one of tetrazodiphenyl a red colouring matter is also formed, and may be precipitated with common salt. One molecule of the disulphonic acid is present in the filtrate. When, however, the substances are mixed in the proportion last described, and warmed, a blue substance is formed, which may also be precipitated by means of common salt. In this case no disulphonic acid is present in the filtrate. The precipitate forms a blue solution with water. In evaporation a bronze-coloured mass is obtained, which also dissolves in water with a blue colour. Alkalis colour the solution red, but hydrochloric acid gives a violet precipitate, which on heating becomes blue. The substance has not yet been crystallised. Its reactions seem to indicate that it is not an ordinary azo colouring matter but a condensation product. Other diparamines of the diphenyl group behave like benzidine, yielding with the above betanaphthol-disulphonic acid azo compounds, which form violet or blue solutions. Among others, diamidofluorene, diamidodiphenyleneketone, diamidodiphenic acid, and diethoxy-diamidodiphenyl may be mentioned. That the blue colour of the compounds is not to be referred to the diphenyl group is shown by the fact that the diazo compounds of orthoamidodiphenyl, para-amidoparabromodiphenyl or paranitrodiphenyl form red colouring matters with the same disulphonic acid. When the betanaphthol disulphonic acid G, or one of the two betanaphthol-monosulphonic acids is substituted for the above disulphonic acid, only red colouring matters are formed. The formation of violet or red products is therefore dependent on the constitution, on the one hand, of the paradiamine, and, on the other, of the beta-naphthol disulphonic acid R.—S. Y.

*Quinoline and Quinaldine.* Dingl. Polyt. Jour. 250, 533. P. FRIEDLÄNDER and C. F. Gohring (Berl. Ber. 1883, 1833) find that the reaction of orthamidobenzaldehyde with acetaldehyde, in presence of sodium hydrate, whereby quinoline is formed, is typical of a large group of such reactions. Thus orthamidobenzaldehyde and acetone, in aqueous solution, react in the cold, on the addition of sodium hydrate, with formation of  $\alpha$ -methyl-quinoline. The process of preparing quinaldines by the action of acetaldehyde upon salts of the primary aromatic amines has been patented (Ger. Pat. 24317, 1882) by the Chem. Fabrik and Actien, vorm. E. Schering. The reaction with aniline is expressed by the equation



The reagents employed are as follow: 100 parts aniline, 150 parts paraldehyde, 200 parts hydrochloric acid, and 3 parts aluminium chloride. These are boiled together from 4 to 6 hours, sodium hydrate is then added in excess, and the bases thereby separated are purified by fractional distillation. Quinaldine thus obtained boils at 240°; orthomethylquinaldine is a liquid boiling at 250°; paramethylquinaldine is crystalline, and melts at 60°. The hydroxy derivatives of the bases are prepared by fusing their sodium sulphonates with four times their weight of caustic soda. The corresponding methoxy and ethoxy derivatives are obtained from the hydroxy compounds by treatment with the respective iodides. Or these may be directly obtained from the anidophenols according to the typical equation



Orthoxyquinaldine crystallises in colourless needles, m.p. 72°; the para compound melts at 213°; methoxy-



quinaldine,  $\text{CH}_3\text{O} \cdot \text{C}_{10}\text{H}_8\text{N}$ , crystallises in shining prisms, m.p.  $125^\circ$ . The quinaldine bases are converted into corresponding hydro bases by reduction with tin and hydrochloric acid. These latter react with the salts of diazobenzene to form red-brown colouring matters; with benzotrichloride to form green colouring matters. L. Kuow (*Berl. Ber.* 1883, 2593) has shown that the product of the reaction of aniline with acetoacetic ether at  $120^\circ$  is converted by treatment with sulphuric acid into oxymethylquinoline. O. Döbner and W. v. Miller (*Berl. Ber.* 1883, 2465) have investigated the hydro base obtained by the action of tin and hydrochloric acid upon quinaldine. This tetrahydroquinaldine,  $\text{C}_{10}\text{H}_{13}\text{N}$ , is a colourless liquid (b.p.  $247^\circ$ ) slightly soluble in water. It is distinguished from quinaldine by the blood-red reaction of its salts in aqueous solution with oxidising agents. It reacts with nitrous acid to form a yellow nitroso compound, whence it appears to be a secondary base. The substitution of the basic hydrogen by an alcohol radical, by treatment with the corresponding halogen compound, gives rise to a tertiary base. Of these tertiary bases the author investigated methylhydroquinaldine, a colourless liquid boiling at  $245^\circ$ — $248^\circ$ . Heated with benzotrichloride and zinc chloride it yields a green colouring matter, similar to malachite green. It is noteworthy that quinaldine under similar treatment yields no colouring matter. Quinaldine further unites directly with the alcoholic iodides to form ammonium compounds. Of these the methyl iodide,  $\text{C}_{10}\text{H}_8\text{NCH}_3\text{I}$ , is remarkable for crystallising in extremely long needles (melting point  $195^\circ$ ). Mixed with the corresponding quinoline derivative it yields on treatment with aqueous potassium hydrate a red colouring matter. From the three isomeric toluidines the authors have prepared the corresponding methylquinaldines. The ortho compound is a colourless liquid (boiling point  $252^\circ$ ); it is reduced by treatment with tin and hydrochloric acid to the hydrobase  $\text{CH}_3 \cdot \text{C}_{10}\text{H}_{12}\text{N}$ , a colourless liquid boiling at  $261^\circ$ . Paramethylquinaldine crystallises in long colourless prisms, melting at  $60^\circ$ , it boils at  $266^\circ$ . The meta compound melts at  $61^\circ$  and boils at  $264^\circ$ .  $\alpha$ -Quinoline carbonic acid,  $\text{C}_{10}\text{H}_7\text{NO}_2$ , is the product of oxidising quinaldine with chloric acid. G. Schultze (*Berl. Ber.* 1883, 2600) has found that on allowing a mixture of aniline hydrochlorate and aldehyde, in aqueous solution, to stand for some time the hydrochlorate of a new base is obtained, which is readily converted into quinaldine by the action of zinc chloride. A similar compound is formed as the product of the reaction of aldehyde with the free base. These results have an important bearing upon the commercial aspect of the treatment of quinaldine. E. Jacobsen and C. L. Reimer (*Berl. Ber.* 1883, 2602) have prepared a yellow colouring matter,  $\text{C}_{15}\text{H}_{11}\text{NO}_2$ , by the action of phthalic anhydride upon quinaldine. Similar derivatives have been obtained from the homologues of the latter, and from picoline. The general formula of these compounds is  $\text{R} \cdot \text{C}_2\text{O}_2 \cdot \text{C}_6\text{H}_4$ . To this class probably belongs the quinophthalone, prepared by Traub (*ibid.* 297) from quinoline obtained from quinquine. Quinoline yellow is regarded by the authors as the prototype of the phthalones. By the action of phthalic anhydride upon paramethylquinaldine, the homologue methylquinophthalone was obtained, crystallising in golden-yellow needles, m.p.  $203^\circ$ . Coal tarpicoline yields two phthalones, probably derive from isomeric picolines. They also dye wool a yellow colour, but are much weaker than the quinoline yellow. In regard to the constitution of the phthalones it is to be observed that they are only formed from the methyl derivatives of pyridine and quinoline, and it is therefore probable that the substitution takes place in the methyl group. On this view the formula of quinoline yellow would be—



W. Spalteholz has also investigated certain colouring matters obtained from coal-tar quinoline. By the action of ethyl iodide this latter is converted into a mixture of ethylquinolineammonium iodide, which may be easily isolated in the form of large yellow prisms, and an uncrystallisable body, which on treatment with aqueous potash gives a much larger yield of colouring matter than the pure iodide. This colouring matter is isolated from the resinous product of the reaction in the form of rhombic

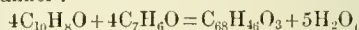
plates, having a high beetle-green lustre, which are soluble in alcohol and in acetone to a carmine solution. The same body was obtained by the action of potash upon a mixture of quinoline and quinaldine-ethylammonium iodides. The process consists simply in dissolving 2 parts of the former to 1 part of the latter in hot water, adding potash and heating for a short time, when the colouring matter separates in the crystalline form, and is obtained pure by washing away a resinous body which accompanies it with ether. The composition of the colouring matter is that represented by the empirical formula  $\text{C}_{33}\text{H}_{25}\text{N}_2$ .—C. F. C.

#### Methylene Blue. *Dingl. Polyt. Journ.* 250, 284.

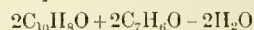
To modify the shade of this colour, which in the pure state is greenish, it is often mixed with methyl-violet or other colouring matter. The admixture is detected by taking advantage of the action of an acid solution of stannous chloride (1 part HCl to 1 part  $\text{SnCl}_2$ ) upon the blue which is to decolorise it completely, whereas the methyl-violet is changed to yellow; so also fuchsin methyl-green and malachite green. On adding sodium acetate the colour is regenerated, and remains unchanged on boiling the solution. The methylene blue, on the other hand, does not reappear.—C. F. C.

#### The Condensation Product of Betanaphthol and Benzaldehyde. W. Trzinsky. *Ber.* 17, 499.

IN a former communication the author made reference to the formation of melinointrisulphonic acid from the product obtained by heating betanaphthol and benzaldehyde with sulphuric acid. This product has been more closely investigated. For its preparation 3 parts of betanaphthol and 1.5 part benzaldehyde were dissolved in 1.5 part alcohol, and treated gradually with 1 part concentrated sulphuric acid. The resulting mass, when recrystallised repeatedly from benzene, chloroform, or carbon disulphide, gave microscopic rhombic plates, melting at  $190^\circ$  to  $191^\circ$  and giving on analysis numbers corresponding with the formula  $\text{C}_{68}\text{H}_{46}\text{O}_3$ . The compound is insoluble in alcohol, ether, and aqueous or alcoholic potash. On heating it with concentrated sulphuric acid it is converted into melinointrisulphonic acid. According to the author's analyses, the condensation of benzaldehyde and betanaphthol is effected in the following manner:—



In the first place, however, the body  $\text{C}_{34}\text{H}_{24}\text{O}_2$  is formed according to the equation—

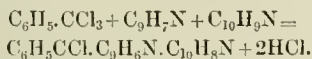


which is converted into the compound  $\text{C}_{68}\text{H}_{46}\text{O}_3$  by the elimination of one molecule of  $\text{H}_2\text{O}$  from two molecules of the first body.—D. B.

#### Colouring Matters from Pyridine and Quinoline Bases. *Dingl. Polyt. Jour.* 250, 466.

ACCORDING to E. Jacobsen (*D. R. P.* 23, 188, 1882), (a) phthalic anhydride yields on heating with the pyridine ( $130^\circ$  to  $230^\circ$ ) and quinoline ( $230^\circ$  to  $310^\circ$ ) bases of coal tar yellow-coloured condensation products. Similar products are in the same way obtained from quinaldine and its homologues—the series of bases prepared by Döbner and v. Miller by the action of paraaldehyde upon toluidine, xylicidine, naphthylamine, etc., in presence of dehydrating agents. The proportions of the reagents are 1 mol. phthalic anhydride to 2 mols. of base, and 1 mol. lime chloride. A temperature of  $200^\circ$  to  $250^\circ$  is necessary to determine the reaction, which is completed in 5 to 6 hours. The colouring base, being insoluble in water, is easily isolated from the resulting mass. It may be converted into a soluble sulpho compound by solution in sulphonyl chloride. The alkaline salts of this derivative are freely soluble in water, and dye silk and wool without mordant. In place of the phthalic anhydride, nitrophthalic acid or phthalimide may be employed. The derivative obtained from these are similar in colour and properties. (b) Processes are described in the D. P. J.

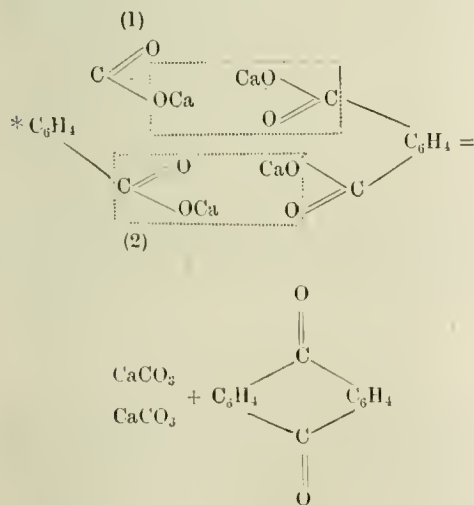
1883, 247, for the preparation of red colouring matters from the quinoline bases by treatment with benzo trichloride, benzal chloride, benzal bromide, and benzal chlorobromide (Ger. Pat. 23, 967, 1882). These bases differ from the majority of tertiary aromatic amines in yielding these colour bases directly, whereas the latter yield leuco bases. The proportions of the reagents are 1 mol. benzal chloride to 2 mol. of the base, together with zinc chloride. After heating 4 to 5 hours at 150°, the product is treated by distillation with steam, for the removal of the residues of the reagents. The colouring matter is then further purified by crystallisation from alcohol or boiling water, and is lastly converted into a sulphonated derivative by the usual method. (c) A mixture of quinoline and quinaldine, or their homologues reacts with benzotrichloride to form violet red colouring matters, the solutions of which exhibit a yellow-green fluorescence. The probable course of the reaction is that represented by the equation—



The quinoline from coal tar (boiling point 230° to 240°), contains 20 to 25 per cent. quinaldine, and therefore requires only a proportionate addition of the latter base in order that the maximum yield of the colouring matter may be obtained.—C. F. C.

#### New Synthesis of Anthraquinone. W. Panaotovits. Ber. 17, 312.

THE observation made by Fittig, and subsequently by Zinke, that anthraquinone is not an ordinary quinone, but a double ketone, is now generally accepted by the majority of chemists. The author has made a series of experiments on the destructive distillation of ordinary potassium phthalate, and has succeeded in obtaining anthraquinone. The formation takes place according to the following equation:—

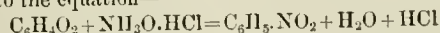


This result confirms the theory which represents anthraquinone as a diketone, and also corroborates Pechmann's views on the formation of anthraquinone (Ber. 12, 2125).

D.B.

#### Nitroso Phenols. H. Goldschmidt. Berl. Ber. 17, (2) 213.

THE reaction between quinone and hydroxylamine in concentrated solution is so violent as to cause the carbonisation of the product. In dilute solutions, on the other hand, the reaction proceeds smoothly, the chief product being paranitrosophenol (*ibid.* 7, 809), formed according to the equation—



Similarly beta-naphthoquinone and hydroxylamine hydrochlorate react with formation of a body,  $\text{C}_{10}\text{H}_7\text{NO}_2$ , which the author has shown to be beta-nitrosoalphanaphthol.—C. F. C.

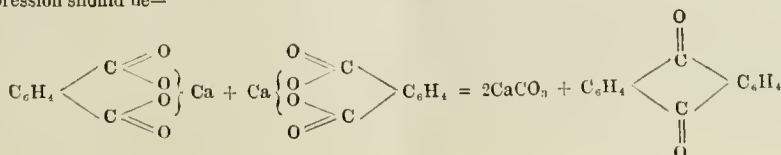
#### Probable Number of Homologues and Isomers of Rosaniline. A. Rosenstiehl and M. Gerber. Ber. 17, 139, and Compt. Rend. 98, 433.

SINCE for the formation of rosaniline in the case of the higher homologues of aniline, the hydrogen of the amido group in the para and ortho positions must be replaced by  $\text{CH}_3$ , the authors conclude that, according to theory, thirteen homologues and (with the isomers) altogether eighty different rosanilines are obtainable, if the hydrogen atom of the  $\text{NH}_2$  group in the meta position is also replaced by  $\text{CH}_3$ . Otherwise only nine homologues and altogether thirty rosanilines are possible.—D. B.

#### On the Dyestuff Rocellin. Dingl. Polyt. Jour. 251, 321.

EMIL ROUSSEL communicates various particulars about rocellin in the *Moniteur de la Teinture*, Nov. 1883, p. 271. This colouring matter, which is prepared by diazotising naphthylaminosulfo acid and pairing with beta-naphthol, is known to be one of the purest of the azo series. Although intended chiefly to replace archil (*Rocella tinctoria*), under certain conditions it can be employed to produce red and crimson colours in place of cochineal and alizarin. Hitherto rocellin could not be fixed on vegetable fibres, and it has chiefly been used for dyeing silk. The employment of it for dyeing wool presented many difficulties at first, owing to the extreme avidity with which the wool absorbed the colouring matter, becoming often unevenly dyed unless special precautions were adopted. Roussel's process is as follows: The bath is slightly acidified with hydrochloric acid, heated to 50° and the wool immersed for 15 to 30 minutes. At this point the rocellin is gradually added, and the temperature raised during half an hour to 90°. The wool is finally allowed to remain another half hour in the bath. Under these conditions the goods are uniformly dyed. By the addition of chrysoin a colour is produced which advantageously replaces turkey-red, and is 50 per cent. cheaper. Ink spots can be easily removed from cloth thus dyed, by oxalic acid, the iron salts having no influence on the rocellin, whereas alizarin red is very sensitive to their action. Different shades can be produced by mixing rocellin with indigo carmine, chrysoin, orange, naphthol yellow, etc. The indigo carmine is added towards the end of the operation, with simultaneous addition of sulphuric acid and glauber's salt. These dyes are as fast as cochineal and much more so than archil. The colour of cochineal and archil changes to yellow under the influence of acids, and to violet-red by the action of alkalis, whereas the freshness of colour of rocellin is not affected by either reagent. The cost of production of rocellin dyes is 80 per cent. less than those made with cochineal, and 40 per cent. less than those made with archil. Since the appearance of rocellin the consumption of archil has considerably diminished.—W. M.

\* This formula, reproduced faithfully from the *Berichten*, is utterly wrong, and so leads to wrong results in the equation. Doubtless the expression should be—

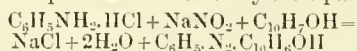


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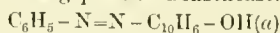


*On the Constitution of the Azonaphthol Colouring Matters.*  
C. Liebermann. Berl. Ber. 16, 2538.

It is generally supposed that the two parallel series of azo compounds, formed by the action of alphanaphthol and betanaphthol respectively on various diazocompounds possess the same constitution, except as regards the position of the hydroxyl group in the naphthol. From an observation communicated to him by Dr. Pfaff, the author was, however, led to an opposite conclusion. Dr. Pfaff finds that when a dilute alkaline solution of alphanaphthol is exactly precipitated with its equivalent of acid, and treated with an aniline salt and sodium nitrite, in the quantities indicated by the equation

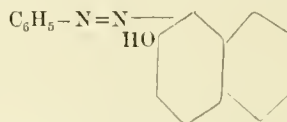


the formation of the colouring matter commences at once. If, however, betanaphthol is substituted for the alpha compound the mixture remains almost colourless; but by the addition of a large quantity of alkali a very beautiful colouring matter is produced. The same difference was observed with other bases than aniline. Although many such compounds have been prepared, the different behaviour of alpha- and beta-naphthol has previously escaped observation, because an alkaline solution of the phenol has always been employed. In their properties the colouring matters from alpha- and betanaphthol differ even more widely than in their mode of formation. Thus Dr. Pfaff made the observation that the alpha compound is easily soluble in cold dilute alkali, while the beta compound is only dissolved to a slight extent in a hot concentrated solution of the same, and is precipitated unaltered on cooling. Hence the two substances must be differently constituted. Similar results were obtained by the author with cumidine and betanaphthylamine, and in all these cases the beta compounds were distinguished from the alpha by their greater tendency to crystallisation, their inferior solubility, and generally by their lower melting points. Benzeneazoalphanaphthol

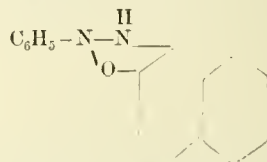


was prepared in a different manner by Typke, though not in a state of purity. The author obtained it, however, in beautiful steel-blue needles by recrystallising two or three times from hot alcohol. The substance melted at 193°, evolving at the same time large quantities of gas, and its solution in concentrated acid possessed a violet-blue colour. Benzeneazobetanaphthol,  $\text{C}_{20}\text{H}_{12}\text{N}_2\text{O}$ , crystallises from boiling alcohol, by rapid cooling in long gold-red, lustrous, leafy crystals, and by slow cooling in long lustrous needles, which possess the colour of cantharidine. It dissolves in concentrated sulphuric acid with a fuchsine-red colour, and it is also soluble in ether, benzene, ligroin, and carbon bisulphide. It melts at about 134°. It is insoluble in an aqueous solution of an alkali, but dissolves readily in sodium alcoholate, being reprecipitated without alteration on addition of water. It possesses weak basic properties, inasmuch as it is dissolved to some extent by concentrated hydrochloric and hydrobromic acids, especially on warming. The hot solution deposits on cooling an unstable green hydrochloride, which gradually gives up hydrochloric acid and becomes red. The alpha compound, on the other hand, is very slightly soluble in these acids. Apparently the alpha compound takes up one molecule, the beta compound two molecules, of hydrochloric acid. The insolubility of the beta compound might be explained by assuming the constitution to be  $\text{C}_6\text{H}_5\text{N}=\text{N}-\text{OC}_{10}\text{H}_7$ , but this is contradicted by its great stability. Small quantities may even be distilled without decomposition. That the nitrogen is really united with the naphthol radical is also proved by the fact that when the substance is reduced with stannous chloride, aniline and amidobetanaphthol are formed. The identity of the latter product with the alphaamidobetanaphthol obtained from betanaphthol orange was shown conclusively by its conversion into betanaphthoquinone by the method of Stenhouse and Groves. Since the substances required for the preparation of benzeneazobetanaphthol are readily obtained, this body may be most advantageously employed in the production of

betanaphthoquinone. The foregoing observations lead to the conclusion that the beta colouring matter possesses the constitution



It had already been observed in the author's laboratory that such substitution products when in the ortho position readily gave rise to anhydro compounds, and the insolubility of the colouring matter in alkalis, as well as the low melting point, would be explained by assuming the formation of an anhydro compound, thus—



It is very possible that the constitution of the sulphonated azobetanaphthol colouring matters differs similarly from that of the alpha compounds. The difference in their solubility in alkalis would, however, be hidden by the presence of the sulphuryl group. The examination of their salts has been so far neglected, and, moreover, especially as regards basic salts, is attended with some difficulty. The constitution of anthrol



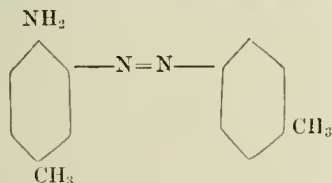
is analogous with that of betanaphthol, and the azo compounds of this substance with diazobenzene and diazocumidine are similarly insoluble in cold alkalis. The difficulty with which the formation of the betanaphtholazo compounds is attended is explained by the fact that the diazo group cannot take the para position in the betanaphthol molecule, but is obliged to take the ortho position. The diazo group thus behaves like other acid radicals, and the general belief that it always occupies the para position is incorrect. A further proof of this fact is afforded by the disazo compounds. Even when the para position is unoccupied the presence of substitution groups in the molecule may cause the diazo groups to take some other position. The separation of isomeric phenol substances may sometimes be effected by precipitating them one after the other as azo compounds by adding different diazo substances to the mixture.

S. Y.

*Orthoamidoazo Compounds.* E. Nölting and O. N. Witt.  
Berl. Ber. 17, 77.

It has hitherto been assumed that by the action of diazo compounds on amines and phenols, amido or oxyazo compounds can only be formed when the para position is vacant—that is to say, the azo group does not attach itself near a side chain. The authors show, however, in this paper that it is possible to obtain compounds where the two groups are attached to neighbouring atoms, i.e., orthoamidoazo compounds. Previous attempts by various chemists to prepare an amidoazo body from paratoluidine have failed, diazoamido compounds only having been obtained, whilst it has been stated that orthotoluidine gives an amidoazotoluene without even the intermediate formation of a diazoamido compound. The authors have found that this latter statement is only true if ordinary orthotoluidine be used, but that if the absolutely pure substance be employed an easily crystallisable intermediate diazoamido compound is obtained. They further find that the failure to obtain an amidoazo compound from paratoluidine is to be ascribed to the use of alcohol as a solvent, and that if, instead, paratoluidine be

employed, the substance can be obtained without difficulty. Diazoamidoparatoluene prepared in the ordinary manner was added from five to six times the quantity of paratoluidine previously melted on a water-bath. One molecule of paratoluidine hydrochloride for each molecule of the diazoamido compound was then added, and the mixture warmed on the water-bath to 65° for ten to twelve hours. The hydrochloric acid is then exactly neutralised with caustic soda, the salt solution removed, and the excess of toluidine removed by steam. The residue is the new amidoazotoluene, which can be purified by crystallisation from alcohol. It forms brilliant orange-red needles, easily soluble in hot alcohol, acetic ether, benzene, etc., and melting at 118.5. Analysis confirmed the formula,  $C_{14}H_{15}N_3$ . Amidoazoparatoluene differs from all other hitherto described amidoazo bodies in giving salts which when solid are pale yellow but in solution are green. The slightest admixture, especially of any other ordinary amidoazo compound, giving red salts, destroys the green colour on treating the alcoholic solution with hydrochloric acid. Apart from this, the new substance, behaves quite like other amidoazo compounds. The hydrochloride crystallises in yellowish-white needles. The authors have prepared the acetyl and benzoyl derivatives, and the sulphonic acid by treatment with fuming sulphuric acid. The disulphonic acid is a yellow colouring matter with a slightly redder tint than the yellow mixture of the sulphonic acids of amidoazobenzene found in commerce. Diazo compounds obtained by the action of nitrous acid on amidoazoparatoluene or its sulphonic acids give red colouring matters with  $\alpha$ -naphthol or its sulphonic acids. By the action of reducing agents the new amidoazo body gives paratoluidine and orthotoluylenediamine, the first example of an amidoazo compound of the benzene series giving by reduction an orthodiamine. It has not been found possible to obtain a quinone or saffranin from the products of reduction, which therefore cannot contain a paradiamine, and in which no metadiamines have been detected. The characteristic ethenyl base of orthotoluylenediamine, on the other hand, can be prepared by means of acetic anhydride. From these facts it follows that the constitution of the new amidoazotoluene is—



The authors have obtained similar compounds from other para-amido bodies, *e.g.*, paratoluidine. With the salts of primary bases amidoazotoluene gives colouring matters of the indulin series, in the study of which the authors are engaged. In connection with the above research a French patent was taken out on May 9, 1882. A German patent has in all instances been refused.—A. S.

#### *Synthesis of Anthraquinoline.* C. Graebe. Ber. 17, 170.

SKRAUP having shown that, by the action of nitrobenzene, glycerol, and sulphuric acid on betanaphthylamine, betanaphthoquinoline is formed, it was thought that on treating anthramine in a similar manner anthraquinoline might be produced. The object of this experiment was to determine whether the body which the author obtained on heating alizarin blue with zinc dust is produced in this case. From his former investigations the author concluded that the constitution of anthraquinoline from alizarin blue is expressed by the following formula:—



Since the amide in anthramine occupies the same position as the nitrogen atom in alizarin blue the possibility

was given that anthramine would yield the same anthraquinoline. On investigation it was found to be the case. On heating anthramine with nitrobenzene, glycerine, and sulphuric acid anthraquinoline melting at 170° was produced. It forms yellow salts, the solutions of which are highly fluorescent.—D. B.

#### *On the Violet Derivatives of Triphenylmethane.* O. Fischer and G. Körner, Berl. Ber. 16, 2904.

It was stated some months ago by O. Fischer and L. German (*Ber. Ber.* 16, 706) that the reduction product of methyl-violet, which melted at 173°, contained no hydrogen in an amido group, capable of substitution, and that it was therefore, in all probability, hexamethyl paraleucaniline. It was also shown that the tetramethylparaleucaniline obtained from paranitrobenzaldehyde and dimethylaniline yield an acetyl compound, which on oxidation gives rise to a green colouring matter. Further investigation has shown that besides the reduction product, which melts at 173°, methyl-violet yields other products, from which acetyl compounds may be prepared. Thus on crystallising the reduction products from alcohol the hexamethyl compound separates first, and the mother-liquor is then found to contain other compounds, which behave like tetramethylparaleucaniline when treated with acetic anhydride, and consequently with oxidising agents. Meanwhile the authors received from Dr. Koch a specimen of a green colouring matter which he had prepared from methyl-violet by treatment with acetic acid, and which proved to be a homologue of the acetyl green described above, for by removal of the acetyl group a violet was obtained which differed from the tetra- and from the hexa- methyl-violet, and was probably the pentamethyl compound. The action of acetic anhydride shows at once whether free hydrogen is still present in the amido group. A small quantity of methyl-violet is warmed with acetic anhydride and sodium acetate, and a drop of the dilute aqueous solution placed on filter paper. The formation of a green rim round the violet stain proves that the violet contains lower homologues of hexamethyl-violet. The violet investigated by the authors was prepared from pure dimethylaniline. It was heated for several hours on the water-bath with acetic anhydride and fused sodium acetate, dissolved in water, and colouring matters precipitated with sodium chloride and zinc chloride. The substance which was thus freed from acetic acid was dissolved in water and fractionated by precipitation with sodium chloride. The unaltered violet came down first, and on reduction yielded hexamethyl paraleucaniline. The green mother-liquor was treated with alkali, and the resulting gray flakey precipitate was filtered, dried, and boiled with absolute alcohol. The alcoholic solution yielded crystals, which after purification melted at 223° to 225°. The mother-liquor contained other products which were not investigated. The analysis of the acetyl compound gave numbers corresponding to the formula  $C_{26}H_{33}N_3O_3$ , or diacetyl-pentamethylpararosanine. On investigation it was found that one acetyl group was combined with nitrogen, the other with the oxygen of a carbinol group. The alcoholic solution when exposed to the air assumed a violet colour, and when warmed with acetic acid yielded the green colouring matter. Unfortunately this substance, though very beautiful, is unstable, and is therefore not of technical value. *Acetyl-pentamethylparaleucaniline* is obtained by reduction of the solution of the green acetyl compound in acetic acid by means of zinc dust, and precipitation with an alkali. It is obtained in colourless needles by recrystallisation from alcohol, which melt at 143° to 144°. Its composition was confirmed by analysis. By oxidation it yields the green colouring matter. *Pentamethylparaleucaniline*: By removal of the acetyl group from the compound last described by boiling with concentrated hydrochloric acid, a product is obtained which crystallises from alcohol in colourless needles. The crystals obtained from its solution in benzene melted at 115° to 116°, and were found to possess the composition  $C_{24}H_{29}N_3$ . The colour of the compound formed by oxidation of this body lies between that of the tetra- and of the hexa- methyl-violet. That the substance is pentamethylparaleucaniline



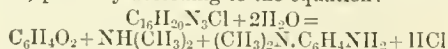
is proved by the fact that it takes up only one acetyl group, and that the acetyl compound differs from acetyl-tetramethylparaleucaniline. The position of the amido groups is the same as in the tetra- and hexa- methyl compounds and in paraleucaniline, because they all yield the same product when treated with methyl-iodide and methyl-alcohol. The analysis alone of the compound would not be sufficient to prove whether the substance contained four or five methyl groups, but the fact that only one acetyl group is taken up decides the question. The explanation of the formation of methyl-violet, proposed by E. and O. Fischer (*Ann. Chem. Pharm.* 194, 295), is in accordance with the results described, but the explanation may now be simplified. It was shown that formaldehyde is produced by the oxidation of dimethylaniline, and it was assumed that the formaldehyde or formic acid united with three molecules of dimethylaniline to form hexamethylparaleucaniline, and that by further oxidation one methyl group was removed, and the pentamethyl compound produced. It is now known, however, that at least two homologous violets are formed by the oxidation of dimethylaniline. The authors now assume that the oxidised methyl groups from the dimethylaniline unite not only with three molecules of dimethylaniline to form the hexamethyl compound, but with two molecules of dimethylaniline, and the molecule of monomethylaniline produced by the reaction, to form pentamethylleucaniline. So also a combination of methane with one molecule of dimethylaniline and two of monomethylaniline would give the tetramethyl compound. The process of formation of methyl-violet is thus analogous with that of Hofmann's fuchsin, and also with that of diphenylamine blue. The great care taken by manufacturers to obtain pure dimethylaniline appears from the above reasoning to be unnecessary. In order to make the process perfect some substance should be employed which would liberate the methane carbon atom, as in Hofmann's fuchsin process. Probably the addition of a benzyl group would then be unnecessary, on account of the pure blue colour of the hexamethyl compound, and because it is only the lower homologues which are acted on by chlorobenzyl. Thus O. Fischer and L. Germann have shown that an alcoholic solution of the leuco base of hexamethyl-violet is not acted on by chlorobenzyl in presence of an alkali, while the authors find that the unaltered hexamethyl-violet is contained in benzyl-violet. *Methyl-green*: The authors find that by removal of methyl-chloride the hexamethyl-violet is formed, and the same substance was obtained from crystal green (the zinc chloride double salt), the mother-liquor containing only traces of other leuco violets. It thus appears that methyl-green is a pure substance, and the facility with which it may be crystallised is thus explained. While it is extremely difficult to crystallise commercial methyl-violet, the zinc double salts both of penta- and hexa- methyl-violet are readily obtained in beautiful needles or prisms.—S. Y.

*Resorcin Blue.* Justus Andeer. *Centrabl. Med. Wissenschaften* 1883, 47.

By heating with egg albumen or with urea (two molecules to one) resorcinol gives a blue sublimate.—A. S.

*Colouring Matters Obtained by the Simultaneous Oxidation of Monamines and Paradiamines.* R. Nietzki. *Berl. Ber.* 17, (2), 223, 227.

The author has investigated the action of ferric chloride upon an acid solution of tetramethyldiamidodiphenylamine and in presence of hydrogen sulphide. The first stage in the reaction is the formation of the green colouring matter  $C_{16}H_{20}N_4Cl$ . This latter then undergoes decomposition, probably according to the equation:—



and the resulting dimethylparaphenylenediamine is converted by the action of the hydrogen sulphide into methylene blue. The author obtained only 5 per cent. of the theoretical yield of the colouring matters, a fact which confirms the view of its being formed indirectly.

The oxidation of paradiamines in presence of primary, secondary, and tertiary bases, is the source of a large number of new colouring matters. Aniline black is probably a member of this series of products. By oxidising paraphenylenediamine in presence of diphenylamine the author obtained a body similar to that which constitutes the green stage of the above. The same substance is also produced, together with quinone, by the oxidation of paramonamidodiphenylamine. Its composition is represented by the empirical formula  $C_{18}H_{12}N_4$ . The close relationship of paradiamidodiphenylamine to the safranines leads to a few observations in conclusion upon this group of compounds. In regard to the empirical formula of phenosafranin the author allows the correctness of that established by Bindschedler (*ibid.*, 16, 864), viz.,  $C_{18}H_{12}N_4$ . At the same time he considers this author in error in assigning 4 atoms of H as necessary for its reduction to the leuco base, 2 atoms H only being required. The error is probably caused in the use of ferrous sulphate, the results obtained with stannous chloride being more trustworthy. The author is engaged in the investigation of the constitution of phenosafranin.—C. F. C.

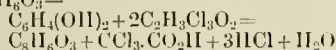
*On 1, 2, 4 Orthoxyldine.* O. Jacobsen. *Ber.* 17, 159.

SOME time ago Wroblewsky described a new xylidine, which he regarded as orthoxyldine [1, 2, 4], without, however, explaining its formation. This substance when treated with nitrous acid yielded a nitroso compound identical with that which the author had previously obtained by the nitration of orthoxylylene. The author has recently prepared chemically pure orthoxylylene on a large scale from coal-tar oils, and having had large quantities of this substance at his disposal, has made an investigation in order to determine whether orthoxyldine [1, 2, 4] described by Wroblewsky is a derivative of orthoxylylene. He found that this substance is not orthoxyldine, nor is its essential constituent this body. Nitro-orthoxylylene,  $C_6H_3.Me_2.NO_2$  [1, 2, 4], is prepared by introducing pure orthoxylylene into eight to ten times the quantity of cold fuming nitric acid. It crystallises from alcohol in long vitreous light yellow prisms, sparingly soluble in cold alcohol. Melting point 29°, boiling point 258°, specific gravity at 30° 1.139. Orthoxyldine,  $C_6H_3.Me_2.NH_2$ , has been prepared from nitro-orthoxylylene by means of acetic acid and iron and tin and hydrochloric acid. It melts at 49° and boils at 226° sp. gr. at 17.5° 1.0755. It is sparingly soluble in cold water, readily soluble in hot water, alcohol, and ether. It crystallises in transparent vitreous rhombic plates, or when cooled gradually in large well-defined crystals belonging to the monoclinic system. Its aqueous solution is not coloured by a solution of chloride of lime. The solutions of its salts impart to firwood a yellow colour. The hydrochloride,  $C_6H_3.Me_2.NH_2.HCl + H_2O$ , forms long, thin prisms, readily soluble in water. Acetoxylide,  $C_6H_3.Me_2.NHAc$ , obtained by the action of acetic anhydride or acetic chloride on orthoxyldine, crystallises from alcoholic solutions in long vitreous prisms, melting at 99°. It is readily soluble in alcohol and hot water. In order to determine the constitution of orthoxyldine the author converted it into the corresponding xylenol, which was found to be identical with the 1, 2, 4, orthoxylenol obtained from orthoxylylene sulphonic acid. Its boiling point was 225°, and it melted at 62.5°.—D. B.

*The Action of Aldehydes on Phenols.* A. Michael and A. M. Coney. *Amer. Chem. Journ.* 5, 349-353.

THIS paper is concerned with the action of aldehydes of the fatty series on phenols. When a solution of equal parts of aldehyde and orcin in 8 parts of alcohol is heated for one minute at 100° with three drops of hydrochloric acid, a bright yellow substance of the formula  $C_{18}H_{20}O_4$  separates out in small round plates, which darken in the air at 100°, are insoluble in the ordinary solvents, and readily oxidise in alkaline solution. Resorcin, in place of orcin, gives likewise, under similar conditions, a compound which, however, decomposes at 100° in a stream of carbon dioxide. When a mixture of

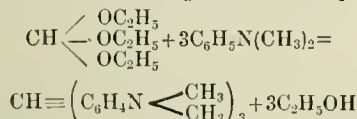
5 parts of chloral hydrate, 10 parts of resorcin, and 40 of water are heated for 12 to 24 hours with reversed condenser, a bright yellow substance separates out, partly before and partly after cooling, which possesses the formula  $C_8H_6O_3$ —



This substance melts with decomposition at a high temperature, is somewhat easily soluble in hot water, extremely so in hot alcohol and acetic acid. It is probably white when pure, but turns yellow at once in the air, crystallises from alcohol in yellow needles, and gives with alkalis a solution which darkens on exposure to the air, whilst by heating with acetic anhydride and sodium acetate it gives the compound  $C_8H_4O_3.(C_2H_3O)_2$ . This separates from alcohol in prisms which melt at  $159^\circ$ , are soluble in hot water, slightly soluble in cold, and moderately so in hot alcohol. The benzoyl compound,  $C_8H_4O_3.(C_6H_5O)_2$ , which is produced by the action of benzoyl chloride on the substance  $C_8H_6O_3$  at  $120^\circ$ , forms faintly reddish needles, melting at  $165^\circ$ , and possessing similar solubility to the acetyl compound. With orcin chloral hydrate gives a substance melting at  $250^\circ$ , whilst its acetyl derivative melts at  $190^\circ$ —A. S.

*Violet Derivatives of Triphenyl Methane.* O. Fischer and G. Koerner. Berl. Ber. 17, 98.

IN this paper the authors describe a new synthesis of hexamethylparaleucaniline by the action of orthoformic ethers or dimethylaniline. If 1 part of orthoformicethyl ether and 3 to 4 parts dimethylaniline be heated for some hours on a water-bath, with the addition of 2 parts of zinc chloride, the following reaction takes place:—



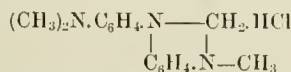
The substance is isolated by freeing the blue-coloured mass resulting from the above operation from excess of dimethylaniline by a current of steam, dissolving in hydrochloric acid, and pouring into cooled ammonia. It separates out in crystals, which, after one crystallisation, appear as beautiful shining plates, which melt at  $172^\circ$  to  $173^\circ$ . Nearly the theoretical yield is obtained.—A. S.

*Preparation of Orthonitrobenzaldehyde.* A. Einhorn. Berl. Ber. 17, 119.

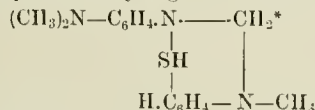
AFTER reviewing the processes previously described for the preparation of this substance, the author recommends the following method, based on the fact that the alkali salts of orthonitrocinnamic acid, when carefully treated with potassium permanganate, give the aldehyde, which, as soon as formed, is absorbed by benzene. 50grms. commercial orthonitrocinnamic acid, suspended in 2½ litres of water, are neutralised with a solution of sodium carbonate, and filtered into a large stoppered bottle, containing 1 litre of benzene. The fluid is kept constantly cool by the addition of ice, and 1,225c.c. of a cold saturated (6 per cent.) solution of potassium permanganate added in small portions at a time, with thorough shaking after each addition. The manganese dioxide which separates is best got rid of by the addition of a warm solution of 155grms. sodium sulphite and sufficient hydrochloric acid to generate the quantity of sulphur dioxide required to dissolve the manganese dioxide, the liquid being kept in motion by a current of air. The fluid now consists of two layers, between which is generally a solid substance consisting of unattacked orthonitrocinnamic acid and orthonitrobenzoic acid. The watery layer may be siphoned off, and the benzene layer filtered and distilled, upon which the orthonitrobenzaldehyde remains as a brown-coloured liquid, which solidifies to a crystalline mass on cooling, and can be dried on an unglazed porcelain plate. The method gives usually from 50 to 53 per cent. of the theoretical yield, which, however, can be increased by using more dilute solutions and adding plenty of ice. It is not proposed to utilise the process commercially, owing to its costliness.—A. S.

*On the Constitution of Methylene Blue.* E. Erlenmeyer. Berl. Ber. 16, 2857. *On Methylene Blue and Allied Colouring Matters.* August Bernthsen. Berl. Ber. 16, 2896.

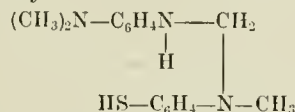
VARIOUS views are held by different observers as to the constitution of methylene blue and violet and the corresponding leuco compounds. Thus Möhlau places the sulphur atom between two nitrogen atoms; Erlenmeyer between a nitrogen atom and a carbon atom of one of the benzene rings; while Bernthsen considers that it is united to a carbon atom of each benzene molecule. Erlenmeyer finds that methylene blue may be prepared from Bindschedler's green by treatment with sulphuretted hydrogen and ferric chloride. In opposition to Nietzki he considers that Bindschedler's green is constituted thus:—



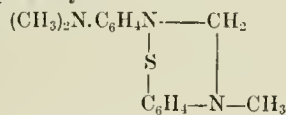
and writes the formula of the compound obtained by the action of sulphuretted hydrogen as below:—



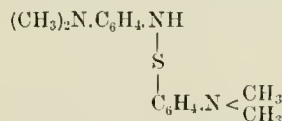
or more probably



Hence the constitution of methylene blue and white would be respectively—



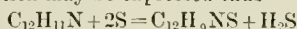
and



Bernthsen attempts to solve the problem by preparing the compound from which the different colouring matters are derived. This compound the author believes to be thioldiphenylamine, to which he gives the constitution



Pointing out the analogy between methylene blue and Lauth's violet, the author suggests that the composition of these substances may be best represented by the formulae  $C_{16}H_{17}N_3S$  and  $C_{12}H_9N_3S$  (or possibly  $C_{12}H_{11}N_3S$ ) in place of those generally adopted, viz.,  $C_{16}H_{19}N_3S$  and  $C_{12}H_{11}N_3S_2$  ( $=C_{12}H_{10}N_3S$ ). According to the author these colouring matters are derived from thioldiphenylamine, the leuco violet being a diamidothioldiphenylamine, and the leuco blue a tetramethyldiamidothioldiphenylamine. The research is not yet completed, but the following results are given in support of the author's view. Thioldiphenylamine,  $C_{12}H_9N_3S$ : Diphenylamine and sulphur are heated together to between  $250^\circ$  and  $300^\circ$  until the evolution of sulphuretted hydrogen has ceased. The product is then distilled and purified by repeated crystallisation from its hot alcoholic solution. The above formula was confirmed by analysis, and the reaction may be expressed thus—

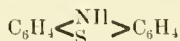


Thioldiphenylamine is deposited from alcohol in the form of light yellow, glittering, leafy crystals. It is also more soluble in hot than in cold glacial acetic acid and benzene, and may be crystallised from these solvents. The sub-

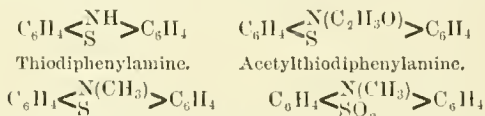
\* The first formula is obviously incorrect, since it would make sulphur a triad and carbon a heptad element.—S. Y.



stance is not a base. It melts at  $180^{\circ}$  (uncorrected), and distils with but little decomposition at about  $371^{\circ}$ . The vapour density, 196.4, confirms the formula  $C_{12}H_9NS$ . Thiodiphenylamine absorbs oxygen from the air, and the surface of the crystals assumes a green colour. Its alcoholic solution, when exposed to the air, becomes at first deep green and finally red, while the addition of ferric chloride produces a green precipitate. Nitrous acid or the fumes of nitric acid colour the solid a deep brown-red. The solution of thiodiphenylamine in cold concentrated sulphuric acid possesses a greenish-brown colour, but in thin layers it appears rose-red. The addition of water produces a crystalline precipitate. Minute quantities of thiodiphenylamine may be detected by means of the following colour reactions. A little glacial acetic acid is first added to the substance, and then a few drops of fuming nitric acid, when a red solution is obtained, which, on addition of water, deposits a yellow nitro compound. On boiling this with an acid solution of stannous chloride a colourless solution is formed, which, after removal of the tin by means of zinc, gives, with excess of ammonia, an intense violet coloration. The addition of ferric chloride to the reduced solution gives rise to a reddish-violet precipitate or a violet coloration. Acetothiodiphenylamine,  $C_{12}H_9NS(C_2H_5O)$ , is prepared by heating the substance for some hours with excess of acetic anhydride. The yield is quantitative. The acetyl derivative crystallises well, and its colour reactions are similar to those of thiodiphenylamine, methylthiodiphenylamine,  $C_{12}H_9NS(CH_3)$ , and ethylthiodiphenylamine,  $C_{12}H_9NS(C_2H_5)$  are formed by the action at  $100^{\circ}$  to  $110^{\circ}$  of methyl alcohol and iodide and ethyl alcohol and bromide respectively on thiodiphenylamine. They both crystallise remarkably well. The methyl derivative melts at  $99.3^{\circ}$ , the ethyl at  $102^{\circ}$ . Thus thiodiphenylamine possesses a hydrogen atom which is easily replaced by alcoholic and acid radicals. That this hydrogen atom is not combined with the sulphur is shown by the chemical properties of thiodiphenylamine, which differ entirely from those of a mercaptan. Hence the hydrogen atom must be combined with nitrogen, and the sulphur must have taken the place of two hydrogen atoms in the benzene rings. The constitution of the compound is therefore most probably—



The oxidation products of the compound were then studied, the methyl derivative being employed, to avoid the presence of the amido group. The oxidation was effected in a boiling aqueous solution of methyl thiodiphenylamine by means of potassium permanganate, and a product was obtained which crystallised from hot alcohol in colourless or reddish crystals, which melted at  $222^{\circ}$ . The composition of the substance was found by analysis to be  $C_{13}H_{11}NSO_2$ . The yield was remarkably good. The substance possesses neither basic nor acid properties, and is unaltered by boiling concentrated alkalis and acids, nor does it give the colour reactions described above. The oxidation product is most probably methyl diphenylaminesulphone, and the constitution of the compounds described is probably as follows—

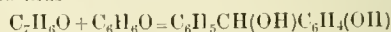


Methylthiodiphenylamine. Methylthiodiphenylaminesulphone. The relationship of thiophenylamine to Lauth's violet and methylene blue is shown by the following observations: The nitration of thiophenylamine with fuming nitric acid takes place easily, and with considerable evolution of heat. The product which crystallises in minute needles dissolves in alkalis with a blood-red colour. By reduction with stannous chloride the colourless tin double salt of the leuco base is formed. Weak oxidising agents convert this compound into a violet colouring matter, which bears a strong resemblance to Lauth's violet, but their identity is not yet determined. When dimethylphenylene green,  $C_{16}H_{19}N_3$ , is reduced

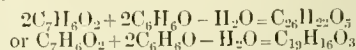
with sulphuretted hydrogen, tetramethyldiamidodiphenylamine is formed, and when the latter compound is saturated with the gas, and then oxidised with ferric chloride, methylene blue is found among the products of the reaction.—S. Y.

#### *On the Condensation of Aromatic Aldehydes with Phenols.* T. Trzeinski. Berl. Ber. 16, 2835.

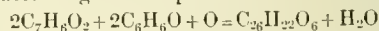
It has been shown by the researches of Baeyer and his pupils that one molecule of the fatty aldehydes (methylal, ethylaldehyde, and chloral) and two molecules of the aromatic aldehydes (benzaldehyde and salicylaldehyde) combine respectively with two molecules of a phenol, one molecule of water being liberated. Baeyer explains the reaction by assuming that an intermediate compound is formed thus—



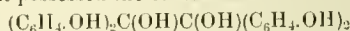
and that two molecules of this compound combine, with liberation of one molecule of water. The more recent researches of Liebermann on the compounds formed by the union of salicylaldehyde and paraoxybenzaldehyde with phenol led to no precise demonstration of the manner in which condensation takes place. The composition of the product obtained from salicylaldehyde did not agree with the formula required by either of the equations—



and Liebermann considered that the colouring matter obtained from paraoxybenzaldehyde and phenol was formed according to the equation—

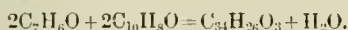


and that it possessed the constitution—

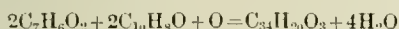


dioxymethylene,  $CH(OH)(C_6H_4.OH)_2$  being first formed and then oxidised by the sulphuric acid. Though the analytical numbers obtained by Liebermann do not agree well with his hypothesis, yet they show that in this case the reaction described by Baeyer does not take place. The investigation of the subject was undertaken by the author, who found that when betanaphthol instead of phenol is warmed with paraoxybenzaldehyde and concentrated sulphuric acid on the water-bath, a very stable orange-yellow crystalline substance is formed, which is the trisulphonic acid of a condensation product, of the composition  $C_{34}H_{26}O_9$ . The compound is best prepared by mixing 5 parts of the aldehyde with 12 parts of betanaphthol, and adding 50 parts of concentrated sulphuric acid. The liquid becomes slightly warm, and the colour becomes at first a deep violet and then red. The mixture is warmed for  $2\frac{1}{2}$  to 3 hours on the water-bath, during which time a slight evolution of sulphur dioxide occurs. The crude product is precipitated by addition of water, filtered, and converted into the easily soluble calcium salt by boiling with calcium carbonate. After repeated crystallisation from hot water the salt is treated with dilute hydrochloric acid, whereby a deep yellow compound of the new substance with hydrochloric acid is formed. By heating in the air-bath to  $120$ – $130^{\circ}$  the hydrochloric acid is removed and the pure substance, to which the author gives the name melinointrisulphonic acid, is obtained. When pure the acid is insoluble in absolute alcohol but moderately soluble in water. In thin layers it possesses a beautiful rose-red colour with a green fluorescence, in larger masses the colour is pure yellow. It is not attacked by boiling nitric acid, and its solutions in this acid and in concentrated sulphuric acid exhibit a beautiful green fluorescence. It is precipitated from its aqueous solution by mineral acids in the form of yellow flakes. An alkaline solution of the acid is reduced by zinc dust, but the compound formed has not been analysed. It is easily oxidised to melinointrisulphonic acid. The acid does not melt at  $300^{\circ}$ , and is stable at that temperature. It is a strong acid, and is not liberated from its salts by dilute acetic acid. It was found on analysis to possess the composition  $C_{34}H_{17}O_9(SO_3H)_3$ . A number of salts were prepared and analysed by the

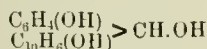
author. Their composition is represented by the general formula  $C_{34}H_{17}O_5(SO_3Me)_3$ . The yield from 5grms. paroxybenzaldehyde and 12grms. naphthol was usually 2grms. melinointrisulphonic acid. Schäffer's betanaphtholsulphonic acid may be substituted for the naphthol; but the acid is not formed by the action of salicylaldehyde on betanaphthol, or of parabenaldehyde on alphanaphthol or on betadinaphthol. It may, however, be prepared, and more economically than by the method described, by heating benzaldehyde and betanaphthol with concentrated sulphuric acid to 200° or 220°. According to Baeyer, when a hot alcoholic solution of benzaldehyde and naphthol is treated with a mineral acid the following reaction occurs:—



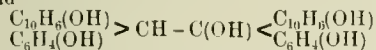
When, however, equivalent quantities of the two acids are heated to 200° to 220° with concentrated sulphuric acid, sulphur dioxide is evolved and melinointrisulphonic acid is formed. In practice it is more convenient to prepare Baeyer's condensation product, and to heat this with the concentrated acid. A solution of 1 part of benzaldehyde and 2 parts of betanaphthol in 1 part of alcohol is treated with 0.6 part concentrated sulphuric acid, and the mixture constantly stirred. The product is filtered, washed with alcohol, and dried at 100°. To one part of this substance 4 parts of concentrated sulphuric acid, heated to 100°, are gradually added. The solution is then heated to 200° in a porcelain basin, when sulphur dioxide is copiously evolved. The acid is then precipitated with water. The formation of the acid may be represented thus—



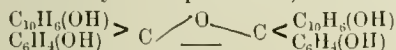
It would appear that at first oxyphenyloxy-naphthyl-carbinol



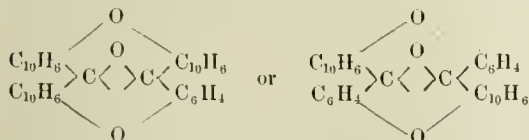
is formed, and that a condensation of two molecules of this substance then occurs, water being liberated. This compound



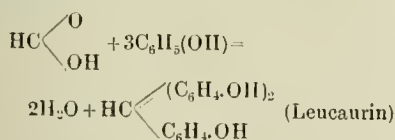
is then oxidised by the sulphuric acid, and the body



is thus formed. The separation of the other two molecules of water might be effected either by the union of two hydroxyl groups with two hydrogen atoms of the benzene or naphthalene groups or with the hydrogen atoms of two other hydroxyl radicals. In the latter case the structural formula of the compound would be either

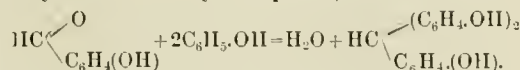


The composition of melinoin shows that the condensation does not take place in the manner described by Baeyer, for three molecules of water are removed, and also two hydrogen atoms are oxidised to water. A similar oxidation was noticed by the author in the formation of a colouring matter from salicylaldehyde and phenol. The formation of aurin and its homologues by heating phenols with formic acid and zinc chloride (Nencki, *Journ. für prakt. Chemie.* 23, 273) may be represented thus—

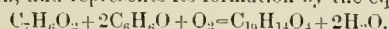


and it might be expected that the same or an isomeric

compound would be formed by the action of salicylaldehyde or benzaldehyde on phenol, thus—



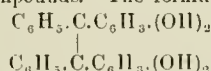
Liebermann prepared a colouring matter by acting on phenol with salicylaldehyde, but found that it differed entirely both from aurin and from rosolic acid. The same compound was prepared by the author, who found its composition to be  $C_{19}H_{14}O_4$ . Since it contains one more oxygen atom than aurin, the author names it oxyaurin, and represents its formation by the equation



S. Y.

*The Action of Aldehydes on Phenols.* Arthur Michael. *Amer. Chem. Journ.*, 5, 338—349.

MIXTURES of aromatic aldehydes with phenols are converted into white resins by the action of quite small quantities of acids, the rate of conversion depending upon the acid used. Thus 20grms. of benzaldehyde and resorcin are converted into resin by one drop of acid. In order to prepare the resin pure, one drop of hydrochloric acid is added to a solution of equal parts by weight of benzaldehyde and resorcin in three parts of alcohol, the mixture filtered when cold, and the filtrate poured into water. The precipitate is washed with water, dissolved in alcohol, again precipitated by water, and dried at 100° in a stream of carbon dioxide or hydrogen. In this way a slightly reddish powder is obtained, which melts with decomposition at 330°, and possesses the composition  $C_{26}H_{20}O_4$ . The undried resin is white, melts under boiling water, dissolves very readily in alcohol, ether, glacial acetic acid, and benzene, and changes rapidly on heating into a dark-brown substance. The colourless solution in alkalis also soon becomes dark brown. The resin dried in a vacuum possesses the composition  $C_{26}H_{20}O_4.3H_2O$ . An amorphous compoundless soluble in alcohol and benzene, and possessing the composition  $C_{26}H_{16}O_4.(C_2H_5O)_4$  is obtained from the white resin by boiling with acetic anhydride and sodium acetate. The resorcinbenzaldehyde resin is converted by the further action of dilute acids into two crystalline products, of which one dried at 100° is isomeric with the resin dried at 100°, but when dried in the air contains  $4H_2O$ . In order to prepare this, a hot solution of 4 parts of benzaldehyde, 10 parts of resorcin, and 20 parts of water is mixed with 3c.c. to 4c.c. of water containing a few drops of hydrochloric acid, heated on the water-bath as long as a precipitate forms, then filtered, treated with acid, and again heated till a precipitate is formed. From this, boiling alcohol extracts, the substance  $C_{26}H_{20}O_4.4H_2O$ , crystallising in quadratic tables or prisms, while the second remains behind in small quantity. The first is moderately soluble in hot alcohol, scarcely soluble in cold alcohol, and insoluble in water and ether. It gives with alkalis a solution rapidly darkening in the air. When boiled with sodium acetate and acetic anhydride it is converted into  $C_{26}H_{16}O_4(C_2H_5O)_4$ , the substance which crystallises from xylene, and is insoluble in alkalis, giving in alkaline solution, by means of sodium amalgam, apparently a reduction product,  $C_{26}H_{22}O_4$ , resembling in its properties the so-called crystallisable resins. Phosphoric acid, and still more organic acids (acetic, formic, and oxalic), cause a slower formation of resin than hydrochloric and sulphuric acids. Experiments with other aromatic aldehydes and phenols showed that the facility with which the formation of resin is caused by small quantities of acids depends upon the nature of both compounds. The formula—



is given provisionally to the benzaldehyderesorcin resin. Fixed alkalis and potassium carbonate likewise produce a resin in a mixture of benzaldehyde and resorcin. The author considers it probable that certain vegetable resins arise from aldehydes and phenols which come in contact with the cell contents.—A. S.



## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Action of Hypochlorites and Chlorates in Dyeing.*  
Dingl. 251, 336.

J. PERSOZ, of Paris, gives a few particulars in the *Bulletin de la Société Chimique de Paris*, 1884, p. 620, which may serve as an amplification of H. Schmid's paper on Witz's oxidation of cellulose (*Dingl. Jour.* 1883, 250, 271). In order to mordant woven goods composed of different fibres with oxide of iron, some dyers use baths of nitro-sulphate of iron and a warm solution of chloride of lime. The object of the latter cannot be to oxidise the iron salt, as it is already in the state of sesquioxide. It acts partly as precipitant of the hydrate of iron and partly in the manner described by Witz, the chlorine or hypochlorous acid increasing the absorptive power of the fibres in the presence of metallic oxide mordants. The oxide of iron is more completely fixed in this case than when precipitated by an alkali or alkaline carbonate. J. Persoz, sen., proposed a similar process for mordanting with oxide of iron mixtures of vegetable and animal fibres which have not the same affinity for metallic oxides. Equivalent quantities of chlorate of potassium and sulphate of iron are dissolved in the cold, the two solutions mixed together, and the woven material is introduced into the dilute bath, which is gradually heated. The chlorate of iron, produced in the first instance by double decomposition, splits up, precipitating hydrate of iron in the fibre. This mordanting is also favoured by the chlorine oxidation products, which are set free and act on the fibres in a well-known manner. If a piece of woven material composed of silk, wool, dog-hair, cotton, flax, etc., be mordanted in this manner, and afterwards dyed with logwood, the colour will be perfectly uniform. J. Persoz, jun., found that this process for mordanting could not only be applied to textile fibres, but also to other carbohydrates, and proposes the following process for mordanting, for example, potato or grain starch, with oxide of iron and dyeing with Berlin blue. Five parts chlorate of potassium and seven parts sulphate of iron are dissolved separately in cold or lukewarm water. Fifteen parts potato starch are ground with water, and the different solutions mixed together in a vessel and diluted to 400 parts. These proportions give a medium blue. The mixture is slowly heated to 55° with constant stirring. When the starch appears sufficiently coloured it is allowed to cool and settle, and is washed by decantation. The mordanted starch is dyed in a very dilute bath of yellow prussiate of potash, which contains one part of concentrated oil of vitriol to two parts of the latter salt, and is slightly warmed. When the saturation is completed the starch is again washed by decantation. A microscopical examination shows that the starch is uniformly dyed, and has preserved its characteristic structure unaltered. Grain starch exhibits the same properties, but less markedly. It is probable that the salts of alumina, chromium, etc., would behave similarly under given conditions, and thus one would be able to dye mordanted potato starch with logwood, alizarin, and astringent dyestuffs, in different shades. The author says that it was the custom to add a certain amount of chlorate of potassium to the ordinary mordant, which aided its fixation in the fibre. But this has been discontinued, as the mixture did not keep well, and gave rise to premature deposition of insoluble metallic oxide in the dye.—W. M.

*On the Mineral Colours Employed in Calico Printing and the Means of Fixing the same.* By Dr. Robert Bourcart. *Monit. Sc.* (3), tome iv.

The essential quality demanded in colours for dyeing and printing is solidity, both as regards lukewarm or boiling soap water, and also with reference to light. Two main classes of colours can be distinguished, viz., mineral colours and organic colours. An intermediate class is formed by the so-called lakes, these being a kind of coloured salts formed by the combination of metallic

or earthy oxides with organic substances, which are frequently colourless. Mineral colours may be classed in two groups: (1) Those produced by chemical reactions in the fibre; (2) those used ready made, and fixed with glutinous substances like albumen. Under the first heading we have chromium yellows and oranges, manganese browns, iron buffs, steam prussian blue, and some other less important colours. Under heading (2) come albumen colours, ultramarine, Guignet's green, chrome yellow, cinnabars, many browns and yellows, lamp black, and lastly metallic powders, such as gold, etc.

**MINERAL COLOURS PRODUCED IN THE FIBRE.—Colours Produced in the Wet Way, Chrome Yellow, and Orange:** If after producing the yellow chromate of lead by double decomposition the pieces are passed through boiling lime water a fine orange is obtained. When other processes precede treatment with lime water, then the lead salt should be printed in the state of a soluble salt on to the cloth prepared with  $\text{Na}_2\text{SO}_4$ , or else passing through baths of sulphate or phosphate of soda is the plan adopted. Sometimes  $\text{PbCl}_2$  is required to be formed, as, e.g., when working in combination with turkey red, and then a bath of bleaching powder is used to convert the nitrate of lead into  $\text{PbCl}_2$ . In the case of a resist under dyed indigo, the lead is precipitated in the state of hydrate. This hydrate is then converted by means of  $\text{HCl}$  and  $\text{K}_2\text{Cr}_2\text{O}_7$ . For the so-called "vert-solide" indigo is dyed on the chromate of lead yellow. For designs in several colours chromate of lead is used in conjunction with aniline black and all alizarine and aniline colours, both for dyeing on a mordant and for steam colours. However, passage through a boiling concentrated solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  soon spoils delicate colours, and then ready-made chrome yellow is used and fixed with albumen. To prevent chrome yellows from blackening with sulphurous fumes, etc., thicken with gum senegal. Such yellows may be restored by being reoxidised with sodium carbonate and potassium ferricyanide, finishing up, if necessary, with another bath of bichromate. Discharges can be worked with alkalis on chrome yellow, e.g., by printing on  $\text{NaHO}$ —soluble sodium chromate being formed. However, we hardly think that this process, which is quoted from Persoz, is now in use. On the other hand, chrome orange is frequently converted into yellow, especially in connection with block work on vat blues with lead resists. In 1819 Professor Lassaigne showed how to fix lead chromate by precipitation in the tissue. It was first applied in 1820 by the firm N. Köchlin and Brothers, of Mulhouse, for discharges on dyed turkey reds. **Manganese Brown:** This colour is not obtained by double decomposition, but by the oxidation of a lower oxide. The prepare used is the chloride or sulphate of manganese, and after drying  $\text{NaHO}$  is used as the precipitant. A passage through dilute bleaching liquor or else hanging for a short time suffices to develop the brown colour required. This brown or "bistre" has the inconvenient property of not resisting the action of even very dilute acids. For obtaining discharges on the brown reducing agents, such as stannous chloride, are used, and then any capable of being fixed by tin can be worked in. The brown may be got of the required shade either in one operation or else, if dark, by passing through several baths of the manganese liquor. The latter method is much better, since then the shade obtained is more regular. If goods are not well washed in bleaching light spots are sometimes obtained in the brown, and this is due to the presence of traces of acid. A previous treatment with dilute  $\text{Na}_2\text{CO}_3$  before proceeding to form the brown will efficiently protect the latter from such defects. Hartmann, of Münster, near Colmar, first used this brown in 1815, and Mercer, in England, in 1830. Mercer also invented several new styles by combining manganese brown, chrome yellow, and vat blue. **Bismuth Brown:** According to Naquet a mixture of nitrate of bismuth and thiosulphate of sodium does not react as long as the salts are in solution, but when cloth is worked in these solutions and then dried, the brown sulphide of bismuth is obtained in twenty-four hours. This method has not yet come into general use. **Hydrate of Iron:** This is worked as in the case of manganese brown, ferrous sulphate and

caustic soda being used. The brown thus obtained is more or less yellow, and is known as "chamois" or "rouille." Resists and discharges can be worked by means of fixed organic acids or chloride of tin. Persoz gives a curious resist, consisting of green soap: this would probably form compounds preventing the hydrate from being precipitated in the fibre. *Soluble Prussian Blue*: In default of any other colour a Prussian blue discharge is used in combination with turkey red. Although turned brown by soap it resists washing with boiling water. The oxalic acid employed both dissolves the blue and liberates Cl in the bleach vat. D. Köchlin-Slouch first used prussian blue in this manner at Mulhouse in 1811. *Phosphate of Copper Green*: M. Camille Köchlin has invented a process for obtaining the above by printing a mixture of copper acetate, sodium phosphate, and ammonia. The green is brilliant, but not very dark. *Oxide of Chromium*: The greens obtained with this substance are more or less gray, and never very full. Attempts to develop Guignet's green on the fibre have not as yet succeeded, and oxide of chromium can well be replaced by albumen colours. When M. Camille Köchlin, in 1832, first applied it to textile colouring a fine green was got by adding arsenic acid. This is now legally forbidden, owing to the poisonous nature of the compound formed. When the new chrome mordant recommended by M. Horace Köchlin for dyeing "violet solide" is steamed a fine pale green is got on soaping. This mordant is a neutral ammonio-potassic chromate mixed with magnesium acetate.

**MINERAL COLOURS PRODUCED ON THE FIBRE BY STEAMING.**—*Prussian Blue*: This was much used formerly, but now comes in only for certain special styles. It is very fine, but does not stand soaping, and is produced by printing a mixture of potassium ferrocyanide and tartaric acid (or oxalic acid) and steaming. The addition of stannous chloride renders the colour much more brilliant, and acetate of chromium somewhat counteracts the injurious effect of soap. Prussian blue has the peculiar property of going pale when exposed to light, and being restored when kept in the dark. It is used for block-work with white discharges on turkey red; for greens it is combined with Persian berries, which is fixed by chromic acetate, and gives a brilliant yellow when tin is present as well. Prussian blue was discovered at the commencement of the eighteenth century by Diesbach, of Berlin, was first used in textile colouring in 1800 by Jean Michel Hausmann, of Lögelbach (Colmar), and as a steam colour was invented by Hindle, of Sabden, near Manchester. In 1820 the process was introduced to Messrs. Dollfus, Mieg, and Company, at Mulhouse, by Dollfus-Ausset, who brought it from England. *Red Prussiate for Darkening Steam Colours*: By means of this salt a darker shade can be given to certain steam colours, such as chocolate, olives, violets, etc. It can be combined with ceruline green, with "violet solide" with alizarine violet, and in these cases acts as a mordant. With steam aniline black and ordinary aniline black it acts as an oxidising agent. *Steam Cadmium Yellow* was discovered by M. Schmidt, of Rouen. This colour is beautiful but expensive, and is irregular in the intensity of its shade. Its formation depends on the fact that nitrate of cadmium and sodium thiosulphate do not react in the cold, but give yellow cadmium sulphide on steaming. *Persulphocyanogen Yellow*: M. Schmidt treats sulphocyanides of potassium or aluminium as if for aniline black, and thus, on steaming, obtains "canarine." He obviates tendering due to HCl by developing his colour by oxidation with vanadium. Canarine is, moreover, an excellent fixing agent for aniline colours, as it has great affinity for these, and soaping becomes practicable. *Steam Chrome Yellow*: M. Jaquet, of Mulhouse, obtains this colour by steaming a mixture of citrate of lead with chromate of zinc and citrate of ammonia. M. Schmidt steams a mixture of barium chromate and lead acetate, but as the barium chromate is not in solution the action only takes place with difficulty. *Steam Manganese Brown* was discovered by Balanche, of Rouen, and obtained by steaming a mixture of potassium bichromate, manganese chloride, and sodium acetate. Probably potassium chloride and manganese

chlorate are formed. The chromic acid oxidises the Mn and is decomposed into chromic oxide. The HCl in combination with the Mn will displace the acetic acid of the sodium acetate.

**MINERAL COLOURS FIXED BY COAGULATION**:—Albumen is almost the only coagulant employed, and occurs in commerce in two forms—blood albumen and egg albumen. Egg albumen is more expensive, but yields an almost colourless aqueous solution, whereas that got from blood is more of a brown colour, and cannot be used for delicate shades: thus ultramarine blue, chrome yellow, and the vermilions can only be thickened with egg albumen. Guignet's green and the various ferric oxides may be mixed with blood albumen, likewise the mixture of ultramarine and lamp-blacks or indigo, which is used for blueish grays and slates. Attempts to replace albumen by chrome gelatine or other substances capable of being coagulated by heat or certain drugs have all been more or less failures. In presence of moisture albumen becomes insoluble when heated above  $74^{\circ}\text{C}$ , but when dry can be heated more strongly, without losing its power of dissolving in water. Commercial albumen always contains from 15 to 17 per cent. of moisture. Albumen colours are steamed like other steam colours, and may be combined with these, but mixtures of the two varieties cannot by any means be made. Thus we may print ultramarine and steam alizarine violet in the same pattern, but their mixture would never produce a violet. In Alsace it seems that albumen colours have almost fallen into disuse owing to the fact that Mulhouse printers have at their disposal a large variety of brilliant and fast colours, and thus can avoid the trouble of trying to procure good albumen at a reasonable price. In England albumen colours are much used not only for discharges on vat blue, for which indeed they are as well in use in France, but also for steam colours. Much albumen can be saved by using suitable thickeners and adding certain salts, which much increase its coagulating power, e.g., sulphate of zinc. The addition of a small quantity of white arsenic,  $\text{As}_2\text{O}_3$ , will prevent putrefaction from taking place. Albumen colours are mostly strengthened with gum dragon (gum tragacanth), as it is transparent and not voluminous when dried on the cloth. Turpentine is of much use, as it prevents frothing, a defect inherent to all albumen colours. Albumen is also used for thickening colours intended for discharges on vat blue. The principle of this style depends on the fact that a mixture of dilute  $\text{H}_2\text{SO}_4$  and oxalic acid liberates chromic acid from potassium chromate, and at the same time coagulates albumen. Ultramarine cannot be used for semi-discharges, owing to its being too sensitive to acids. Albumen colours can resist soaping if not at a boil and of too long duration, but they are much damaged by friction, and hence should be soaped at full width, as is mostly done in England. Dollfus, Mieg, and Company first employed these colours in 1844, and also fixed ultramarine by means of the whites of fresh eggs. Already in 1820 Broquette and Blondin, of La Glacière, near Paris, fixed lapis-lazuli or natural ultramarine by means of albumen, and Gonin, of Rouen, used it about the same date for fixing lamp black. Albumen discharge colours are of more recent date, and were introduced by Camille Köchlin in 1875, being now largely employed. In 1876 M. Depierre devised a curious method of obtaining albumen discharges on vat blue. Potassium ferrieyanide and sodium carbonate are steamed with albumen. The indigo is destroyed by the oxidising action of the ferrieyanide in alkaline solution, and the albumen is simultaneously coagulated. This method is, however, we believe, not in use, and is merely given as a scientific curiosity. In time, perhaps, chemists will succeed in replacing egg albumen, and trials are being made to obtain colourless blood albumen. Although this has not yet been done completely, still certain makers have arrived at very satisfactory results. Several chemists are at present studying the manner of fixing colours by means of chrome gelatine, and the process used consists in preparing the cloth with a mixture of bichromate of potash and sodium thiosulphate, the colours being thickened with gelatine. Another substitute for albumen is lactarine, which is prepared from milk and is



one of the constituents of cheese. Solution is obtained with ammonia, and on desiccation the lactarine becomes insoluble. This substance is difficult to work with, and is hardly used at all now. Some printers use a solution of caoutchouc in light oils or benzine. This is rather too expensive a process, and ordinary gelatine is preferred. Gelatine is printed, and whilst still damp the gold or bronze powder is dusted over, the pieces are then dried and calendered. Albumen colours are almost always mineral. Ultramarine is one of the most important, and is used in different shades, ranging from violet to pure blue. Green ultramarine is the incompletely calcined substance, and there are light and dark shades. Light shades of good covering power are got by letting down the blue with zinc white. For dark shades, such as prussian blue, the ultramarine is mixed with indigo ground with water, and sometimes blueish grays are got with ultramarine and lamp black. Much ultramarine is also used for blueing and finishing, and sometimes the goods are chlorid and blued at the same time. *Guignet's Green*: This colour was patented in 1858 by Guignet and Salvétat, and prepared by heating a mixture of potassium bichromate and boric acid. In 1868, Kestner took out a patent for making it by heating chromic hydrate with boric acid. Guignet's green is not as brilliant as the Schweinfurt colour, but is non-poisonous and resists acids and alkalis completely. *Chromium Yellow and Orange*: As albumen colours these are much used, and possess much brilliancy and fullness of tint. When steamed in a closed space, however, they turn brown, this action being due to the sulphur, which is a constituent of albumen. If any ultramarine is in the neighbourhood of the yellow, the latter is sure to be completely spoiled. If 2grms. to 3grms. of cadmium nitrate per litre be added to the colour, it is found that CdS, which is of a fine yellow colour, is formed in preference to PbS. *Yellow Lake of Persian Berries*: This is one of the few vegetable colours fixed by albumen. It is obtained by precipitating the extract with a salt of tin. The alumina lake, which has a somewhat different shade, is also sometimes prepared. These are fine yellow, not tarnished by sulphur, and can be mixed with ultramarine for various shades. Besides being used as albumen colours, these lakes are also employed for tin discharges on manganese brown, either alone or else admixed with eosine. Alizarine lakes have been patented by Jacobsen for printing, but they are too expensive, and the same remark applies to the so-called ammoniacal cochineal. *Vermilion or Cinnabar*: This red is much used, but is rather dull when compared with alizarine and cochineal red. Sometimes eosine is added to it for shading purposes, and the bluest qualities are the most esteemed. It resists acids very well, and has not been employed very long in printing. Other red colours are the red oxide and the red chromate of lead. The latter is probably what is known and much used in England as "muck vermilion." *Ochres and Oxides of Iron generally*: By means of oxides of iron more or less hydrated and heated to different temperatures a series of colours is obtained ranging from yellow to red and passing through the browns. These can be used for mixing with each other or else with different colours. They are sensitive to acids, and are fixed by means of blood albumen. Light grounds done with these oxides never fade by the action of light, which is the case with alizarine and woads. *Lamp Black*: This was the first colour fixed by albumen. It is much used for mixtures, for the grays it gives alone are not first-class, being too yellow. Good slates are got with lamp black and ultramarine blue. *Coal*: M. Camille Köchlin ground up coal in a mill with water as if for indigo. On printing with albumen it seems he obtained very fine blacks. The coal must be picked, not all kinds being equally suitable. This colour has the advantage of not interfering with others when printed in a pattern of several colours, which is not the case with aniline black.—H. A. R.

*On Modern Dyeing Processes.* Dr. Reimann. Monit. Se. (3) tome xiv.

ALTHOUGH the improvements made during the last twenty-five years have been to a certain extent brought

about by the introduction of aniline colours, yet the most important and most interesting of recent advances do not consist in methods of fixing new colours, but in intelligent amelioration of old methods, in the simplification of receipts and processes, and in the utilisation of well-known colours in new shapes and forms. The introduction of the new colours was much facilitated by the fact that they could fix themselves on animal fibres without the use of any mordant. At first, indeed, wool and silk were dyed directly, but after a while experience indicated the desirability of abandoning this primitive mode of procedure. Cotton soon proved difficult to deal with, as this fibre cannot be dyed with aniline colours in well-nourished shades, unless it be first suitably prepared and mordanted, and this preparation and mordanting varies for different colours. Years of practical trials have been required for determining the mordants most suitable for each colour. As is well known the aniline colours proper, e.g., fuchsine, Paris violet, etc., are bodies of basic properties, and have been fixed in two manners, which plans may be termed general methods. Either the fibre has been as it were mechanically prepared by the fixation of a porous material such as starch, or  $\text{SiO}_2$ , these being capable of attracting and retaining the colour, or else a substance of acid character is employed to unite with the colouring base, and form an insoluble compound. Shades obtained by the use of starch or  $\text{SiO}_2$  are very fugitive, and this method has been almost given up in favour of the plan of producing on the fibre insoluble salts of the colour base. The most important of these salts are those formed with fatty acids, and the tannates. The former are costly, as the dye-bath is but very incompletely exhausted, hence the tannin mordants have become the most usual nowadays. It is curious that cotton, although of very different composition to animal fibre, takes up almost as much tannin as the latter, and when this cotton is subsequently passed through the dye-bath, the shades obtained are well nourished, and fairly solid, owing to the formation of a tannate of the colour base. Recently the stability of many shades has been much increased by rendering the tannin insoluble before passing the cotton through the dye-bath, this being done by means of the tannate of antimony. Any soluble salts of antimony may be used, but tartar emetic is found to work best. With tannin this yields a precipitate of little known composition, which is formed without difficulty, and is fixed intimately. It is true that objections have been made to antimonial mordants on sanitary grounds, but we must remember that only a small portion of the Sb in a dyed fabric is in the soluble state. Further, Sb has in these cases not rarely been taken to be present on the strength of the indications of Marsh's apparatus. The public prejudices against aniline colours on wool and silk are no doubt to a large extent due to the fact that at first these fibres were dyed without mordants at all, thus obtaining shades which were certainly beautiful but extremely fugitive. Further, uneven shades were also found to result, and of course this was due to the great affinity of the animal fibres for this class of colouring matters. This disadvantage was counteracted by the addition of soluble salts, e.g., Glauber's salt. However more was required, colours were demanded capable of standing soaping and fulling, and the aniline colours have not these desirable properties. In the end means were found of making the anilines resist soaping, and this was done in the following manner: A salt was added to the dye-bath capable of producing with  $\text{CO}_2$  or fatty acids an insoluble compound. Such a salt is found in  $\text{MgSO}_4$ . When the cloth is run through a bath of soap or  $\text{Na}_2\text{CO}_3$ , the soluble alkaline salt produces a soap or else insoluble  $\text{MgCO}_3$ , and the alkali, soda, or potash, transformed by double decomposition into neutral sulphate, has no longer any injurious effect on the colouring matter. By replacing  $\text{MgSO}_4$  by  $\text{ZnSO}_4$ , as has been done lately, resistance to fulling is also attained. A considerable step in advance was the dyeing of silk in the soap-bath, by which means the pristine brilliancy of the fibre, to which much of its value is due, is retained. When eosine appeared on the scene, the dyer became acquainted with a new class of colouring matters. Silk is dyed in an old soap bath, and wool in a bath slightly

acid with acetic or sulphuric acids. When the fibre to be dyed is impregnated with a body very finely divided and capable of fixing colouring matters, it is found that the shades come up much better nourished and more stable. Sulphur furnishes the most curious example of this method of mordanting, and it is used at the present time in dyeing wool with aniline green, a bath of hyposulphite of sodium being used and acidified with  $\text{H}_2\text{SO}_4$ . For fixing eosine the milk of sulphur is directly added to the dye-bath. Eosine was not easily fixed on cotton, but this was done at last with alumina. The idea that eosine, especially in combination with fluoresceine, which, as far as appearance goes, can be used for dyeing cochineal reds on wool, would ever drive out the natural colouring matter was effectually dispelled by the return from the East of a consignment of these goods. It was soon found that eosine cannot stand the action of sunlight. At present almost the only use for eosine is in producing certain delicate reddish-green dichroic shades on silk, wool, and cotton. The so-called azo colours are daily becoming of greater importance. Both the acid and the basic group are represented here. The colours range from green-cast yellow, to red-cast violet, the series passing through orange and ponceau. They are fixed either in an acid bath or else by the production of an insoluble salt, *e.g.*, of alumina. The ponceau series have latterly put cochineal much into the shade, as is well shown by the fact that a brand selling six years ago at four shillings can to-day be obtained for eightpence. The importation of cochineal has been reduced by one half, and millions of marks formerly leaving the country now remain at home. For military purposes the azo colours cannot at present replace cochineal, owing to the effect of sunlight, although they stand soaping and fulling much better. Silk dyeing and the production of "mode" colours have been much assisted by the azo dyes. Instead of the expensive archil we now use an azo-ponceau at a lower price and more stable. Picric acid, which is loose and bleeds on mere washing, is replaced by an orange or an azo yellow. Thus "mode" colours, green-cast olive, various tinted browns, "faded" shades, etc., are obtained more cheaply, more stable, and of superior appearance. Hence "mode" dyeing has much increased, and the disappearance of archil is but a question of time. We must now sketch out the manner in which time has affected methods of fixation for natural colouring matters known during ages. Amongst these the redwoods are included, employed only a few years ago to produce red and ponceaux shades on cotton, wool, and silk. As an example of the primitive method of proceeding, the wool-dyer, before passing the fibre into the red-dye, used to expose it to the air and sun for at least fifteen days! This state of things has, however, become incompatible with the present development of industry, and redwoods are merely used for producing certain mixed colours. Lac-dye, which played a great role in former times, is now practically out of the market. Safflower has been replaced by safranine and madder, later still by alizarin. We have not yet got substitutes for certain vegetable colours which are still employed either for particular shades or else for the sake of great cheapness. An example of this class is found in turmeric. Black dyeing is one of the most important branches of textile colouring, and since time immemorial blacks have been obtained with salts of iron and crude tannin. Later on logwood was introduced, and the method remained pretty much the same until 1850. Then the oxide of chromium began to be employed as a mordant, and chrome black, especially used for wool, was got by logwood and chrome baths. By the latter plan loading of the fibre is much reduced, and hence the wool remains elastic. Economy is also attained as the logwood pigments are almost entirely utilised. During the last few years "mode" dyeing has undergone some interesting improvements, principally in the direction of simplification. Since the introduction of aniline colours, in which we have intense colouring power, together with very small weights, the dyer has become exceedingly nice about vegetable colours, and requires them to be presented to him in as highly a concentrated state as possible, *viz.*, in the state of

extracts. No cotton dyer now prepares his own logwood liquor, but wool dyers prefer preparing their own logwood baths, and they are in the right. The cause, however, does not rest with the logwood but with the consumer's endeavours to get cheap drugs. Many refuse to pay the minimum price of pure logwood extract, and are then supplied with a concoction of more or less logwood together with chestnut extract. This is all right for cotton, where black dyeing, as a rule, is carried on with iron, but wool is more frequently dyed on chrome, and the black got is less intense when the logwood has been let down with chestnut extract, and the tannin in the latter renders the wool hard. Under the name of hematine, a French house is selling a pure logwood extract, and this gives great satisfaction in dyeing wool. Amongst other wood extracts used in large quantities, we must mention quercitron, or bark extract, which is imported from America under the name of flavine. Tannin is prepared in large quantities, in an almost pure state, by many manufactories. Formerly it fetched a high price, but now can be bought at 99-100 per cent. for about eightpence per pound. Nowadays, with the use of the pure acid, much more certainty is obtained in mordanting. One colouring matter, known for ages, and very important in its original form, but much more so in its present one, is madder. In times gone by extracts of madder were prepared, and endeavours made to isolate the various colouring matters from the root, but now these researches, which have borne ample fruit, are only of historic interest, since artificial alizarin has quite put out madder. Formerly about three months were required for dyeing a Turkey-red, and afterwards this time was reduced to six weeks. Owing to great improvements made in the oil prepares for Turkey-reds, *e.g.*, sulpholeates, sulphoricinates, etc., and attributed by some to Müller-Jacobs, and by others to Storck, the operation can now be finished in twenty-four hours. The process of mordanting with oil is found beneficial in other cases besides that of Turkey-red. Indigo dyeing has also not remained untouched by a quarter of a century's universal progress. In ancient times the hot vat was the one most employed, but it was found that the constant watching it required was a great disadvantage, and that the proper regulation of the fermentation process was an operation of exceeding nicety, and moreover a very risky one. Cotton dyers more frequently employed the copperas and lime cold vat, but the frequent losses of colour were the stumbling-block here. In 1850 the dyer Leonhard used zinc dust as the reducing agent in an indigo vat, and soon after observed that this blue colouring matter dissolved with great ease in an aqueous solution of  $\text{SO}_2$  previously treated with zinc. Schützenberger and Lalande went further in this direction, and the economical and convenient vat bearing their name was the fruit of their experiments. It can be worked on the small scale almost as well as on the large, this being by no means the case with other vats. On the same principle Kallab based his process for extracting indigo from the waste of indigo-dyed cotton or woollen cloth.

H. A. R.

#### *Dyeing Cotton Hanks with Aniline Black in the Cold.* Dingl. Polyt. Jour. 251, 425.

A. REXARD\* describes a process for the production of aniline black on cotton fibre without the aid of heat. In the ordinary method the hanks to be dyed are placed in a cold bath, having for example the following composition: for 50kg. cotton, 800 litres water, 5kg. aniline, 20kg. hydrochloric acid  $21^\circ \text{B}$ , and 7kg. bichromate of potassium, and as soon as the colour begins to develop it is heated to  $50^\circ$  or  $60^\circ$ ; the operation is generally finished in 1 to 3 hours. In the new process the same chemicals are used, but in different proportions. The fol-

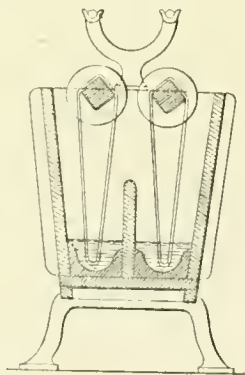
\* Vide A. Renard: *Traité des matières colorantes, du blanchiment et de la teinture du coton.* (Paris, 1883, J. Baudry.)



lowing are recommended as the best. For 100kg. cotton :

	Kilos.
Hydrochloric acid 21° B.....	16 to 20
Sulphuric acid 66° B.....	20
Aniline.....	8 to 10
Bichromate of potassium.....	11 to 20
Sulphate of iron.....	10

The quantity of water used is much smaller than in the old process, as the reaction has to take place in a proportionately shorter time and without the aid of heat. On this account the dye vats have a peculiar form. Each vat is 2 metres long, the shape, as the figure shows, admitting of the smallest possible quantity of solution being employed. The hanks are turned round by means of the four-cornered reels fixed over the vat. Generally several vats are placed end to end to enable all the reels to be rotated at once by an endless chain. This arrangement effects a considerable saving in hand labour, and prevents the workman's hands from coming in contact with the chromic acid solution; also, owing to the regular motion of the hanks, uniform oxidation takes place. It is also better to use a mixture of hydrochloric and sulphuric acids, as hydrochloric acid alone gives a bluish-black, sulphuric acid alone a reddish-black, but the two together the nearest approach to an absolute black colour. The quantity of bichromate has to be increased, as the action of the chromic acid is less energetic in the cold than at a temperature of 50° or 60°. The presence of sulphate of iron is not absolutely necessary, but it helps to increase the stability of the black colour. In practice the aniline is mixed with the hydrochloric acid, previously diluted with an equal volume of water, to prevent the aniline salt from crystallising out; then the



sulphuric acid, similarly diluted, is added, and finally a solution of the sulphate of iron. The bichromate is dissolved in another portion of water. The hanks, after having been boiled, washed, and wrung out, are placed on the reels, and the solution in the vat having been sufficiently diluted with water, and the half of the bichrome solution added, the reels are rotated for 1 to 1½ hour. The goods have now a black colour, and at this point are removed from the vat; the remaining half of the bichromate solution is added; and then the dyeing is proceeded with as before, until the right tone is produced, generally after 1 to 1½ hour. The dyed goods are now washed and boiled with a solution containing 5grms. of soap per litre, and about 2 per cent. of carbonate of soda. Soap alone gives a violet or red black, carbonate of soda alone gives a bluish colour, and makes the cotton hard, therefore both are used, producing the best black and a soft fibre. The black made in this way is sufficiently pure for all purposes, is unaltered in the air, and does not become green even when exposed to the action of dilute sulphurous acid. Bronze black is produced in a similar way, by washing and drying the unsoaked cotton; blue black by treating the soaped goods with carbonate of soda.—W.M.

*Apparatus applicable to Bleaching Keirs, etc.* J. K. J. Foster, Bolton. Eng. Pat. 3351, July 6, 1883. Provisional protection only.

By means of an ejector or otherwise a vacuum is formed in the chamber below the false bottom which supports the cloth, and the percolated liquor is forced by suitable means up the uptake pipe, the circulation of the liquid being thus expedited.—W. E. K.

*Dyeing Cotton Fabrics.* F. A. Gatty, Accrington. Eng. Pat. 3896, August 11, 1883.

For the purpose of dyeing fabrics chrome yellow the cloth is impregnated with a soluble lead salt, and whilst still moist is passed through a box containing gaseous ammonia, in order to fix oxide of lead on the fabric. The colour is then raised in the usual way by passing through a solution of a soluble chromate or bichromate.—W. E. K.

*Fixing Aniline Colours upon Fibrous Materials composed of either Animal or Vegetable Fibre.* Law Heppenstall, junior, Milnsbridge, Huddersfield. Eng. Pat. 3522, July 17, 1883.

IN treating cotton the fibre is first prepared in separate baths of sunaek and double muriate of tin. For wool and silk this preliminary treatment is not essential. Cotton is dyed in a bath containing oxalic acid and sulphate of copper, together with the aniline colour. For wool and silk the aniline colour is first added to the dye-beck, and then, at a later stage, bichromate of potash in the case of wool, and red argol, alum, and green copperas in the case of silk.—W. E. K.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Detection of Free Sulphuric Acid in Aluminium Sulphate.* Dingl. Polyt. Journ. 250, 283.

MILLER has shown (*Berl. Ber.* 1883, p. 1992) that for this test, by the titration method, methyl-orange is the best indicator, since, although its colour is changed to orange by normal aluminium sulphate, a trace of free acid causes a further distinct change to red. The ultramarine and the logwood tests are shown by the same author to be not sufficiently delicate.—C. F. C.

*On the Reactions in the Sulphuric Acid Chambers.* G. Lunge and P. Naeff. Chem. Industrie, 1884, 5-19.

THE experiments were conducted in the chambers of the works of Schnorf Brothers, in Uetikon, and of the British Alkali Works at Widnes. It was found that in a system of chambers working normally the active oxidising agent is nitrous acid. Nitrogen tetroxide only occurs when there is a large excess of nitrous fumes, and in the farthest part of the system of chambers where the reducing action of the sulphurous acid is very weak, and where the chamber gases are deficient in sulphuric acid, which, as is known from Winkler and Lunge's experiments, retards the oxidation of nitrogen above the trioxide. The quantity of oxygen has no influence on the formation of the tetroxide. The nitrous fumes from the Gay-Lussac towers contain only the trioxide. The loss of nitre in the works at Uetikon amounts to 2 to 3 per cent. of the weight of sulphur. The authors show that 46 to 70 per cent. of this loss is due to incomplete absorption in the Gay-Lussac towers, and that it is not caused by reduction to nitrogen monoxide. The hot gases entering from the Glover towers do not rise at the top of the chambers and gradually sink on cooling, but mix thoroughly at once in the nearest part of the chamber. The oxidation of the sulphurous acid is therefore very energetic as far as the middle of the first chamber, after which it decreases. The composition of the chamber gases in a vertical section of the first chamber was found to be very uniform, a slight excess of sulphurous acid and oxygen being found in the middle portions.—S. Y.

*Rapid Estimation of available Chlorine in the Materials employed in Bleach Works, Paper Works, and the like.* R. Baur. *Dingl. Polyt. Journ.* 251, 173, 1884.

IN an extended inquiry, made by the author at the instance of the authorities of Württemberg, into the condition of the bleaching industry, special attention was paid to the methods of testing the raw materials and chemicals in the different stages of the processes, and it was soon revealed that a rapid and reliable method, easily worked by an intelligent foreman, for the estimation of available chlorine, was a desideratum. The tests usually made for the control of the strength of bleaching liquors consist of an areometric reading with a Gay-Lussac or Twaddell hydrometer, and the determination of the number of degrees of indigo oxidised by a given quantity of liquid. The extreme crudity of the first method will be at once admitted, especially as the relation between specific gravity of, and available chlorine in, a liquor varies with every variation in the quality of the raw material. Titration with standard solution of indigo certainly is supposed to give a measure of the available chlorine, but the author has not found the method reliable, and, owing to the first colour change being a gradual one, different observers, and even one and the same observer at different times, may fail to obtain concordant results. The author proposes first of all to express all analytical results in terms of 1 milligramme of available chlorine per litre of liquor, so that a practical indication is at once provided without lengthy calculation. As regards the analytical process, the author has introduced Wagner's modification of the Dupasquier-Bansen iodine method into several works, and has found it, in the hands of an intelligent foreman, to answer its purpose admirably. A solution of sodium hyposulphite is prepared, by titration, of such strength that 1 c.c. corresponds to 1 milligramme of chlorine. Solutions of potassium iodide and hydrochloric acid are also required. About 10 c.c. of bleaching liquor are carefully measured in a graduated tube, and a few cubic centimetres of the solution of potassium iodide added, until a maximum degree of turbidity and brown colour be reached. Hydrochloric acid is then added until the turbidity vanishes, and the clear brown solution titrated with the above solution of sodium hyposulphite, until the brown colour suddenly changes to the clear blue of ordinary water. The number of cubic centimetres of hyposulphite solution employed, multiplied by 1,000, and the product divided by the number of cubic centimetres of liquor taken, gives the number of milligrammes of available chlorine in 1,000 c.c. The quality of ordinary bleaching powder may be determined by dissolving 5 grms. in 1,000 c.c. of water, and titrating as before. The author has found Penot-Mohr's method, depending upon the oxidation of arsenious acid, equally rapid and accurate, but from the poisonous nature of the reagent hardly so suitable for factory work.—W. D. B.



*Production of Soda Crystals by the Ammonia Process.* C. D. Abel. *Eng. Pat.* 3856, 1883.

IF the temperature of a solution of bicarbonate of soda of suitable density be raised to boiling point, the bicarbonate will be transformed into sesquicarbonate. If a quantity of boiling water be now added, and violent ebullition with stirring be brought about, about 80 per cent. of the excess of  $\text{CO}_2$  will be driven off, and the solution may be concentrated to between  $30^\circ$  and  $40^\circ$  B., and the monocarbonate crystallised out. The remaining bicarbonate is used in succeeding operations, or may be decomposed by lime or sulphuric acid before crystallisation. The ammonia and carbonic gas evolved during the process are refrigerated and utilised.—H. J.

*Extraction of Sulphur Compounds from Alkali Waste.* Jas. Simpson, Liverpool. *Eng. Pat.* 3624, July 24, 1883.

YELLOW LIQUOR is pumped into a large cylindrical vessel and air blown through it from the bottom. When sufficiently oxidised it is run into a suitable false-bottomed vessel with a quantity of alkali waste and treated with steam under pressure. When sufficiently digested the

liquor, containing now almost all the sulphur compounds of the waste in a soluble form, is run off, and may either be treated directly with muriatic acid for the precipitation of sulphur and the generation of sulphuretted hydrogen, or, after partial oxidation, for the precipitation of sulphur only. Before treatment with acid the liquor must be freed from hydrate of lime by subsidence and decantation. In case of a deficiency in the supply of the yellow liquor, some of the finished liquor from the digesting apparatus or a solution of sulphurous acid may be used in its stead.—A. R. D.

*Treatment of Liquors Produced in the Ammonia Soda Process, and obtaining Hydrochloric Acid therefrom.* L. Mond, Northwich. *Eng. Pat.* 3820, Aug. 4, 1883.

THE object of this invention is to utilise the chlorine of the valueless by-product chloride of calcium in the manufacture of soda by the ammonia soda process, and to obtain therefrom hydrochloric acid. To effect this the solution of ammonium chloride resulting from the operation of carbonation, instead of being treated with lime as heretofore, is evaporated, and after the removal of the sodium chloride, which salts out, treated with sulphuric acid in sufficient quantity to form the acid sulphate of ammonia. It is then heated to  $250^\circ$  F., HCl being given off. This is condensed by any known method. The acid sulphate, either before or after complete evaporation, is neutralised with ammonia or a salt of ammonia, and neutral sulphate thus obtained. Gas liquors may also be utilised in this way after treatment by the ammonia soda process.—C. C. H.

*Manufacture of Hydrochloric Acid from Chloride of Ammonium, etc.* L. Mond, Northwich. *Eng. Pat.* 3922, Aug. 13, 1883.

THE first part of this invention deals with obtaining hydrochloric acid from ammonium chloride. If this salt be treated with sulphuric acid in the usual way, partial decomposition only is effected, and to remedy this the patentee adds sufficient sulphuric acid to form the acid sulphate of ammonia, and heats to  $250^\circ$  Fahr., when the whole of the HCl is given off without loss of ammonia. This is condensed by any well-known process; the solution of acid sulphate is either neutralised by ammonia or a salt of ammonia and evaporated to dryness, or else first evaporated to dryness and the solid salt broken up and treated with gaseous ammonia. In either case neutral sulphate of ammonia is the result. The second part of the invention relates to the formation of manures rich in ammonia. The acid sulphate of ammonia is mixed, by grinding with water, with bone meal, phosphorite, etc., in such proportions that the excess of acid in the salt above that required for the neutral sulphate is sufficient to render all the insoluble phosphate present soluble. The resulting product is dried and ground.—C. C. H.

*Manufacture of Precipitated Phosphate of Lime, and Recovery of Sulphur from Alkali Waste.* Walter Weldon, Rede Hall, Burslow. A communication from Emile Lombard of Marseilles. *Eng. Pat.* 3884, Aug. 10, 1883.

By this patent it is proposed to precipitate phosphate of lime from solution in hydrochloric acid by the addition of calcium sulphhydrate. It is also proposed to use this process in connection with the recovery of sulphur from alkali waste as follows: A solution of sulphhydrate of lime is to be treated with a hydrochloric solution of calcium phosphate in a closed vessel, which is, however, provided with an outlet for the sulphuretted hydrogen disengaged in the operation. Half of this sulphuretted hydrogen is to be treated for the recovery of sulphur, and the other half passed into a mixture of alkali waste and water for the production of a further supply of calcium sulphhydrate. The vessel in which the precipitation was effected contains thereafter a solution of calcium chloride, holding the precipitated phosphate in suspension.

A. R. D.



*Treatment of Vat Waste.* Hartley Kenyon. Eng. Pat. 3524, 1883.

SELECTED vat waste is dried in a retort, decomposed with superheated steam, and discharged into a finishing furnace. The  $\text{SH}_2$  evolved is treated with ammonia, or with galvaniser's flux liquor or chloride of zinc. The charge in the finishing furnace is treated to obtain caustic lime or carbonate lime.—H. J.

*Improved Glover Tower.* M. A. Walsh. Eng. Pat. 1486, 1884.

To secure a better distribution of the sulphuric acid descending the Glover tower, the same is constructed internally with a gothic or lancet arch in place of the usual round arch. To obviate the discolouration of the acid by the bricks ordinarily used they are replaced by blocks of quartz.—H. J.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Theory of Cement Hardening.* Dingl. Polyt. Jour. 251, 431.

E. LANDRIN calls the silica which has been precipitated from a silicate by an acid and ignited at a red heat "hydraulic silica." It is this which effects the hardening of hydraulic mortars. It abstracts such a quantity of lime from the lime water in the course of a few days that the mass corresponds to the silicate  $4\text{CaO} \cdot 3\text{SiO}_2$ . Gelatinous silica obtained by decomposing soluble glass with hydrochloric acid and soluble silica obtained by analysis behave in the same way. The above compound of hydraulic silica with lime is named by Landrin "Puzzo-Portland." This is the main constituent of all hydraulic mortars, not the silicate  $2\text{CaO} \cdot \text{SiO}_2$ , as assumed by Le Chatelier. The theory of Le Chatelier, that the hardening of cement depends, as in the case of gypsum, on supersaturation phenomena, is disputed by Landrin. W. M.

*Improvements in the Manufacture of Plaster-of-Paris.* J. Tomlinson. Eng. Pat. 3438, 1883.

AN iron or copper tube rotating in a furnace chamber receives the ground gypsum from the mill, roasts it, and delivers it into a receiving chamber, where it is cooled and bagged.—H. J.

*Production of Marble.* J. Heineman. Eng. Pat. 3614, 1883.

THE article manufactured from gypsum is heated to from  $100^\circ$  to  $120^\circ$  Celsius, dipped in  $\text{CaCl}_2$  solution, and then in hot concentrated  $\text{MgSO}_4$  solution. The pores are closed by the sulphate of lime formed whilst the  $\text{MgCl}_2$  is removed by washing. The article is then treated alternately with solutions of gelatine and of tannin. To produce coloured marbles solutions of metallic salts are used.—H. J.

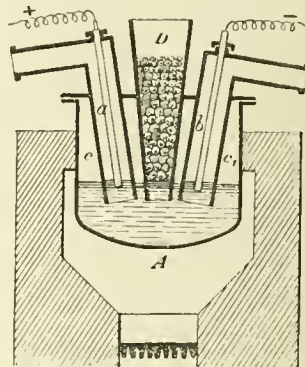
*New or Improved Refractory Material.* W. R. Hutton, Patrick, N.B., and A. Grainger, Cardross, N.B. Eng. Pat. 3528, July 18, 1883. Provisional protection granted only.

STEATITE or soap stone and fire clay are reduced to a fine powder mixed into a paste with water in a pan mill, the mass so obtained moulded into bricks, pipes, or other articles of the desired form. The proportions vary according to the nature of the purpose to which the articles are applied, and where a higher degree of refractoriness is desired powdered plumbago to the extent of one-third of the weight of the compound may be used with advantage. C. C. H.

## X.—METALLURGY, MINING, Etc.

*On the Employment of Electricity in Chemical Industries.* Ferd. Fischer. Dingl. Polyt. Journ. 251, 418.

R. P. HERMAN (Ger. Pat. 24682, April, 1883) proposes to precipitate zinc electrolytically from dilute solutions of sulphate of zinc with the help of sulphate of the alkalis or alkaline earths (potassium, sodium, ammonium, strontium, magnesium, aluminium) added, singly or mixed together. These additions appear only advan-



tageous when one has solely to deal with dilute solutions of sulphate of zinc. According to M. Kiliani (*Berg. und Huttenmännische Zeitung*, 1883, p. 251), during the electrolysis of a solution of sulphate of zinc, 1.33 specific gravity, the anodes and cathodes consisting of zinc plates, the evolution of gas is greatest with a weak current, diminishes with an increasing current, and ceases when on 1 square centimetre pole surface 3 milligrammes zinc are precipitated per minute, as the following table shows:—

Intensity current in mg. zinc per minute and 1 sq. cm.	Gas evolved in c.c. on 15grm. zinc precipitated.
0.0145 .....	2.10
0.0361 .....	2.27
0.0755 .....	0.56
0.3196 .....	0.13
0.6392 .....	0.33
3.7274 .....	—
38.7750 .....	—

The precipitate obtained with a strong current was very firm. From a 10 per cent. solution the precipitate was best with a current of from 0.4 to 0.2 milligramme zinc. From very dilute solutions the zinc was always obtained as a sponge, accompanied by energetic evolution of hydrogen. With a weak current and from a 1 per cent. solution oxide of zinc was also precipitated, even with an electro-motive force of 17 volts, when only 0.0755mgrms. zinc per minute and 1 square centimetre cathode was precipitated. The size of the pole surfaces has consequently to be adjusted according to the strength of the current and the concentration of the solution. The process of Blas and Miest for the extraction of metals from their ores appears good. It is based on the fact that the natural sulphur compounds conduct electricity when they are heated and pressed into plates. Such plates are used as anodes and cathodes. For working zinc blende, the bath consists of sulphate of zinc, and the following reaction takes place:  $\text{ZnSO}_4 + \text{ZnS} = \text{Zn} + \text{S} + \text{ZnSO}_4$ . The blende does not require to be roasted, and the sulphur is obtained as such, and a weaker current is required than in the older processes. When lead glance is to be worked, a bath of nitrate of lead is employed. It is calculated that for 1<sup>st</sup> per hour 2 kilos of lead and the corresponding quantity of sulphur are obtained. A similar process is that of E. Marchese (Ger. Pat. 22429, May, 1882), the chief difference being that the ores are filled into iron baskets and suspended in the precipitation tank. A piece of metal connected with the positive pole is laid on the surface of the ore. The proposal to use the ores partly as anode and partly as cathode is new. According to Marchese, if the anode be made of iron pyrites and the cathode of copper pyrites, the

sulphur from the copper pyrites which is evolved as sulphuretted hydrogen, and the sulphur from the iron pyrites which becomes combined as protosulphate of iron, can be usefully employed, the copper being simultaneously precipitated on the cathode. The electrolytic extraction and refining of copper is now of great importance. According to Siemens and Halske, of Berlin, there are five machines  $C_1$ , each with 12 baths, in action in the Oker Works. The oldest machine has now run for four years, day and night. Each machine works with 4 to 5c. (water power), and precipitates per cell and hour 1 kilo. copper—that is, about 300 kilos. daily. By making the baths double the size and doubling their number, the same machines can still work them with nearly the same working power, and precipitate double the quantity of metal. The Norddeutschen Raffinerie, Hamburg, produces with six No. 1 Gramme machines, and one Wohlwill machine 25,000 kilos. copper daily. Oescher and Mesdach, in Biele, have one Gramme machine built according to Wohlwill's directions, which yields 800 kilos. copper per day. Hilarion Rowe, in Marseilles, has a 5c. No. 1 Gramme machine, with 40 baths and 900 square metre anode surface; the cathodes are only 0.5mm. thick, and are 5c.m. from the anodes. The machine makes 850 revolutions per minute, requires 240 kilos. coal daily, and yields, with 8 volts and 300 amperes, 250 kilos. pure copper. Keith's process for refining lead has been adopted by the Electro-metal Refining Company in New York. The bath consists of a solution of sulphate of lead in acetate of soda; gold, silver, and antimony remain on the anode. When the proper conditions are observed the lead extracted is pure, as the following analyses show:—

	Raw Lead.	After Electrolytic Precipitation.
Lead .....	96.36	99.9
Silver.....	.544	0.00068
Copper.....	.315	0
Antimony.....	1.07	trace
Arsenic.....	1.22	trace
Zinc and iron.	.4886	0

For the production of potassium and sodium P. Jablockhoff fills the heated boiler A with the chlorides of potassium or sodium, which are to be decomposed through the funnel D. The electrodes are introduced by the knee pipes c and c. The chlorine gas evolved from the positive pole a, and the metal set free at the negative pole b are continuously removed.—W. M.

#### *Treatment and Utilisation of Tin and Lead Dross and of the Slag resulting therefrom.* T. Lloyd, Aberdylais. Eng. Pat. 3350, July 6, 1883.

THE cakes of scum or oxide formed on the surface of molten tin or lead, or a mixture of both, are placed in the furnace and exposed to a degree of heat a little above the melting point—then stirred or rabbled until the residuum has assumed the form of powder which is termed dross. Having taken the dross out of the furnace a little water is mixed wherewith to prevent the minor particles from escaping up the flue. Then are intimately mixed therewith carbon, carbon and lime, and alkali, which substances are obtained by utilising certain by-products, such as soot or powdered coke, impure chalk, and the by-products from nitric acid works. To one ton of the dross are added—of alkali compound (powdered) 1cwt., of powdered coke or soot  $\frac{1}{2}$ cwt., and of waste or impure chalk  $\frac{1}{2}$ cwt., also in a powdered state.—J. T.

*An Improved Manufacture of Nickel and Cobalt from their Ores.* C. D. Abel, London. Communicated by A. Krupp, Bernsdorf, Austria. Eng. Pat. 1464, Jan. 15, 1884.

THIS invention relates to a process for smelting nickel and cobalt in such a manner that the cast metal shall be not only perfectly malleable and ductile but shall also be compact and free from air bubbles. Molten nickel and cobalt absorb both carbon and oxygen. On solidifying the greater part of the oxygen passes off, and the porous metal produced is mostly unmalleable on account of the greater or smaller percentage of carbon contained therein. The above defects are obviated by impregnating

the metal with manganate or permanganate of potash or soda, or with other manganates or permanganates. For this purpose pure oxide of nickel or cobalt in the form of cubes is reduced at a moderate heat. The somewhat porous pieces thus obtained are then impregnated as much as possible with a solution of manganate or permanganate containing not more than 4 per cent. of the salt. The pieces are then dried, and are best smelted in crucibles in a furnace with air blast, and at a very high temperature. In order to remove the oxygen introduced into the molten metal by the salt there is introduced into it, at the moment when the mass becomes liquid, or a little later, some black flux, obtained by heating tartar to redness, and some charcoal. In place of the black flux aluminium or calcium-zinc, produced by Caron's process, may be used in combination with charcoal.—J. T.

#### *Treatment of Complex Ores for the Separation or Extraction of the Metals Contained therein.* W. P. Thompson, Liverpool. Communicated by G. T. Lewis, Philadelphia, U.S.A. Eng. Pat. 3652, July 25, 1883.

THIS invention has for its object the treatment of complex and refractory ores containing lead, zinc, and copper, with very often antimony, arsenic, and other base metals, besides a valuable quantity of silver and gold, together or singly. Such ores at a low temperature form only a sluggy waxy mass, whilst at a high temperature the bulk of the metal, including much of the gold and silver, instead of smelting down, will be carried off as fumes. The best way to treat these ores is to use as high a temperature as possible with a highly-oxidising atmosphere, so as to volatilise them as completely as possible, thus separating them from the matrix and the non-volatile metals. The fumes thus obtained are condensed by forcing them through a series of cooling pipes, and afterwards straining them through flannel or other fibrous bags, or other means of condensation and separation, from the gaseous products of the furnace in which the fumes of volatilised metals are suspended. An ore consisting chiefly of sulphides of lead, zinc, and antimony, with silver and gold, is best treated by being subjected, whilst in a fine state of division, to a heated blast of air without the admixture of fluxes, fuel, or the gaseous products of combustion. In the case of refractory complex cupreous ores, containing much silica and other impurities, it will be found best to employ a cupola furnace supplied with a heated blast of air at the bottom, using also a mixture of coal, coke, or charcoal, and fluxes. An experimental plant which has proved very successful, is as follows: First, a low cupola furnace, with feed opening for ore and fuel, and sloping bottom; second, a box into which the metal is tapped, while the slag passes into a water-box; third, a water-back tyure for the blast; fourth, four blowers to blow in the heated blast, carrying the fumes formed in the furnace into the hood hereafter mentioned; fifth, a hood which receives the gases, and passes them on to the cooling pipes; sixth, the cooling pipes and exhausting fan; seventh, a sheet-iron receiving bin, where the solid fumes collect, while the gases pass out through the interstices of (eighth) a bag or bags. Most of the lead, gold, and silver collect in the bin. The remainder of these metals, with most of the arsenic, zinc, and antimony are found in the bags, whilst most of the copper is obtained as regulus in the box aforesaid.—J. T.

## XIV.—AGRICULTURE, MANURES, Etc.

### *Phospho-Mining in Canada.*

THE Canadian correspondent of the *Liverpool Journal of Commerce* says: The Minister of Agriculture states officially that the amount of phosphates shipped from the port of Montreal, during the past calendar year was, in round numbers, 19,000 tons. This industry in its production materially benefits the country generally, as the men employed at the mines, the teams that haul it from the places of production, the freightage of it by rail, the ocean freight, etc., all represent a certain amount of capital expended by this agency. It is expected that 24,000 tons will be shipped from Canada to Great Britain during 1884.



*Experiments with Stable Manure and Artificial Manures.* L. Guillaume. Biedermann's Central-blatt. für Agrikulturchemie 12, 512. Annales Agronomiques, 1883, vol. 9, No. 1, 30-36.

THE experiments were carried out on an estate in Harancourt (Ardennes), and on the land of the Horticultural School, at Villepreux, Seine-et-Oise. The land at Harancourt had been well manured for many years, but was poor in phosphates, and therefore afforded a good test of the value of phosphates and superphosphate. The land at Villepreux contained but little lime, but was rich in nitrogen. The results are given in the tables. The author concludes that in the case of corn the increase of crop caused by the addition of phosphate to the stable manure is not great enough to cover the expense. The quantity of maize was not increased by the phosphate, though the superphosphate acted beneficially. In the case of potatoes, the opinion of Déherain, that it is better to use no stable manure, is confirmed. The crops of turnips was but slightly increased by phosphates or superphosphate. The author agrees with Déherain, Corenwinder, Pagnoul, and Ladureau, that it is not economical

tie rods, and by means of nuts and springs a continuous pressure is exercised upon the cover and hence upon the contents of the silo. The claim is for this mode of applying pressure to wooden or other silos.—J. M. H. M.

*Improvements in the Treatment of Blood to prepare it for use as a Manure or for other purposes.* W. G. Strype, Wicklow. Eng. Pat. 787, January 5, 1884.

THE claim is for adding sulphate of iron, alum, etc., in solution to blood, in defecating the latter, and preparing it for manure.—J. M. H. M.

## XV.—SUGAR, GUMS, STARCHES, Etc.

*Improvements in the Manufacture of Compounds of India-rubber, Gutta-percha, and like Materials.* H. A. Huth. Eng. Pat. 1571, Jan. 16, 1884.

VARIOUS processes have been from time to time devised for attaining the object of this invention, viz., to reduce the cost of articles manufactured from indiarubber, gutta-percha, and analogous products by combining with them

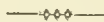
TABLE A.—AT HARANCOURT.

Manure Employed.	Oats.		Potatoes.			Maize for Fodder	Schleswig Turnip	Corn.	
	Straw.	Grain.	Sound.	Unhealthy	Total.			Straw.	Grain.
	kg.	hl.	kg.	kg.	kg.	kg.	kg.	kg.	hl.
None	5000	61'35	18000	none	18000	37200	38400	5050	19'43
15000 kg. stable manure	5850	72'50	18000	1000	19000	55200	40400	5200	20'14
15000 kg. stable manure+1000 kg. phosphate.	6000	74'25	17000	1200	18200	55200	41200	5800	21'18
15000 kg. stable manure+1000 kg. superphosphate.	6300	75'00	17850	350	18200	61200	45000	6000	23'96

TABLE B.—AT VILLEPREUX.

Manure Employed.	Oats.		Potatoes.			Maize for Fodder	Turnips (Mammoth).	Soy.	Corn.	
	Straw.	Grain.	Sound.	Unhealthy	Total.				Straw.	Grain.
	kg.	hl.	kg.	kg.	kg.	kg.	kg.	kg.	kg.	hl.
None	3600	53'55	22050	4610	26700	55000	80000	80000	4050	32'20
15000 kg. stable manure	4200	55'75	22050	4450	26500	100000	100000	100000	4500	33'25
1000 kg. phosphate	5000	56'00	24750	3000	27750	65000	111000	111000	4600	33'75
1000 kg. snperphosphate	5050	55'33	14630	2800	18530	67000	110000	111000	4850	33'85
500 kg. Chili saltpetre	5150	55'10	30770	2520	33290	82000	155000	90000	4200	33'00

to employ phosphates or even superphosphate on previously well-manured land. Stable manure was found to answer very well for maize at Villepreux. Turnips and potatoes were greatly improved by treatment with Chili saltpetre.—S. Y.



*Treating Insoluble Phosphates.* H. J. Haddan. Eng. Pat. 3687, 1883.

PHOSPHATE rock is powdered, mixed with powdered dolomite, and calcined. The carbonic acid of the dolomite is driven off, the iron and alumina of the phosphate rock converted into insoluble oxides, and the phosphoric acid remains combined with lime and magnesia.—H. J.

*An Improved arrangement of Silo, and Press in connection therewith.* S. H. Stocks, Cleckheaton, Yorkshire. Eng. Pat. 3857, August 8, 1883.

THE floor, sides, and cover of the silo are constructed of wood, and iron tie rods are secured to beams in the floor and pass upwards through the silo to the outside of the cover. A screw thread is cut upon the upper part of the

resins and gums of a more or less brittle nature. The inventor attributes the uniform failure of these processes to the neglect of attention to two facts—first, that the resins and gums used have melting points differing amongst themselves and from those of the indiarubber and gutta-percha with which they are to be combined; secondly, that the resins and other materials all contain volatile oils, which, if not removed, cause the finished product to be spongy and imperfect. To overcome the first difficulty, the inventor takes admixtures of gum kauri resin, amber, colophony, gum anisé, copal, lac, sandrac, mastic, and such analogous resins, and combines them, with or without earthwax, in such a way that the combination has a melting point corresponding with the temperature to which the indiarubber, with which it is to be combined, will be exposed in the manufacture. Thus, if the goods are intended to be "cured" at 240° Fahr., the combination of resins, etc., must have a melting point of 240° Fahr. To remove the volatile oils the mixture is heated to 20° Fahr. above its melting point till no more fumes are evolved. In practice the following combinations have been used: No. 1. Earthwax, 2; resin (colophony), 4; kauri, 8; amber, 5. No. 2. Earthwax, 1; colophony,

2; kauri, 8; shellac, 4. No. 3. Earthwax, 4; colophony, 6; kauri, 16. No. 4. Earthwax, 2; colophony, 2; kauri, 9. No. 5. Earthwax, 2; colophony, 2; amber, 6; shellac, 6; kauri, 4. If sulphur is used, it is found that the mixed resins added have the effect of permanently fixing this body. With these materials, or with indiarubber and sulphur alone, the inventor also combines insulite.—A. J. K.

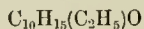
*Apparatus to be Employed in the Refining of Sugar.* C. E. van Haesendonck. Eng. Pat. 3403, July 10, 1883.

THE object of this apparatus is to shorten the time occupied in the purifying and drying of sugar in moulds. Fig. 1 is a side elevation and vertical section; Fig. 2 is an end view of the same apparatus. It consists of a series of vertical screws C about 10in. long, hollow throughout their length, and working in suitable nuts B fixed to a solid slab A. To the top of each screw is attached a funnel E communicating with the central perforation of the screw, and intended to receive and support the sugar mould F at its tapered end. At a suitable height above the screws is a horizontal pipe H, from which descend vertically a number of short side branches, one corresponding to each screw axis. Each of these branch pipes is provided with a strong wide flange, covered with an indiarubber washer, and the flanges are connected with the slab A by strong iron supports by means of hand wheels against the indiarubber washers, and so make good joints. The wash liquor and hot or cold air or gas can now be forced through the sugar through the pipes L, M, N, O, each of which is provided with a cock. By this apparatus and a pressure of 5 to 10 atmospheres, the sugar can be purified and dried much more rapidly than by the ordinary method. Fig. 3 is an enlarged detail of the screw C.—A. J. K.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

*On Matieo-Camphor.* K. Kügler. Ber. 17, 2841.

THE leaves of *Piper angustifolium Ruizet Pavon*—matieo leaves—yield about 2·7 per cent. of an ethereal oil, which is dextro-rotatory, and the greater part of which distils over at 200°. The residue on cooling deposits crystals of matieo-camphor, which melt at 103° (Flückiger). The author has recently investigated this substance. He found that the crude camphor melts between 83° and 103°, and after several recrystallisations at 94°. The purified product is readily soluble in alcohol, ether, chloroform, benzene, and petroleum spirit, and is free from the strong odour and camphorous taste which the crude oil possesses. On treatment with hydrochloric acid it assumes a violet colour, which rapidly changes to blue, finally to green. On analysis numbers corresponding with the formula  $C_{12}H_{20}O$  were obtained. The author is of opinion that further researches into the properties of matieo-camphor will lead to interesting results. It may prove to be the ethyl derivative of ordinary camphor—



D. B.

*On Caffeine, Xanthine, and Guanine.* Emil Fischer and Ludwig Reese. (Ann. 221, 336-344), Berl. Ber. 16, 3071.

THE authors prepared their derivatives from chloro- instead of bromo-caffeine. The chloro compound was prepared by passing dry chlorine gas into a solution of carefully-dried caffeine in chloroform, until the evolution of hydrochloric acid had ceased. From the chloro-caffeine diethoxyhydrocaffeine was prepared, and this was reduced with hydriodic acid to hydroxycaffeine, which, when treated with phosphorus oxychloride and strongly cooled, yielded beautiful crystals of a substance of the composition  $C_{10}H_{13}ClN_4O_4$ , chlorine apparently taking the place of an oxethyl group. Alcohol dissolves the substance with reformation of diethylhydroxycaffeine,

and water converts it into dimethylalloxan. Amalinic acid is easily oxidised by chlorine in presence of water to dimethylalloxan. When distilled, amalinic acid yields, in addition to pungent smelling vapours, either cyanic acid or an ether, and a compound  $C_{12}H_{14}N_4O_8$ , which is difficultly soluble in water, cold alcohol, and ether, but easily in chloroform, glacial acetic acid, and in alkalis. It melts at 260°, sublimes at the same temperature, and distils with partial decomposition when more strongly heated. It reduces an ammoniacal silver solution when warmed, and when evaporated with nitric acid yields dimethylalloxan. The authors give the compound the name desoxyamalinic acid. The methyl ether of guanine could not be prepared by the action of methyl iodide on the lead or silver salt. Guanine, when treated with bromine, yields bromo-guanine,  $C_5H_4BrN_5O$ , a crystalline powder, which decomposes without melting. It is almost insoluble in cold water, alcohol, and ether, and dissolves with difficulty in hot water. Its hydrochloride loses hydrochloric acid at the ordinary temperature. Bromo-guanine dissolves in alkalis and ammonia, and its solution in these substances gives crystalline precipitates with silver and lead salts. The silver and lead salts, when treated with methyl iodide, appears to yield a small quantity of the methyl ether, bromo-caffeine being also formed. Bromo-caffeine is converted by nitrous acid into bromo-xanthine,  $C_5H_3BrN_4O_2$ , which may also be prepared by the bromination of xanthine. It resembles bromo-guanine.—S. Y.

## XXII.—ANALYTICAL CHEMISTRY.

*Presence of Arsenic in Glass as a Source of Error in the Detection of Arsenic.* W. Fresenius. Zeit. Anal. Chem. 22, 397; and Ber. 17, 2938.

As arsenious acid and arseniferous materials are often used in the manufacture of glass, the resulting glass frequently contains arsenic. The author has made a series of experiments with Bohemian glass (containing 0·20 per cent. arsenic), Thuringian glass (containing 0·08 per cent. arsenic), and a refractory glass (containing a trace of arsenic), the object being to study the influence which this impurity may have in the various methods for determining arsenic. It is stated that in testing for arsenic its presence in the glass apparatus employed must not be overlooked, otherwise the most important mistakes may at times be made. Alkaline solutions being capable of taking up arsenic from glass should not be heated in glass vessels. This is not, however, the case with acids. The presence of arsenic in glass is not likely to lead to error in the Marsh test. (The author attributes the browning of glass when heated to arsenic rather than to lead.) In the Fresenius and Babo process it is of more importance. If, as is customary, the mixture of potassium cyanide and soda is brought into direct contact with the glass, the results are vitiated should the glass contain arsenic. It is therefore recommended to place the mixture in a porcelain boat introduced into a tube of glass as free as possible from arsenic. The contents are cautiously dried before a high temperature is applied. The results are then perfectly reliable.—D. B.

## New Books.

ANALYTICAL SUMMARIES OF THE PATENTS, DESIGNS, AND TRADE MARKS ACT, 1883, AND OF THE PATENT LAWS OF ALL FOREIGN COUNTRIES AND BRITISH COLONIES. By A. M. and WILLIAM CLARK, Fellows of the Institute of Patent Agents. London: A. M. and William Clark, 53, Chancery Lane, W.C. 1884. 8vo. volume, bound in cloth, with preface, introduction, and subject matter, covering 197 pages, and an alphabetical index occupying about 10 pages. THE work commences with an analytical summary of (Great Britain and Ireland), with comments thereon.



(Great Britain and Ireland), with comments thereon. Then follow analytical summaries of the patent laws of foreign countries. In the case of the foreign countries, the matter in the text proper is commenced, and headed with summarised details in small type of the geographical extent and position of the country, its population, export and import values, colonial possessions, chief industries and their centres, weights and measures, and coinage, etc. Then, headed with date of formation of existing patent law, follows in bold type the summary, arranged in paragraphs, each paragraph commencing with title of the subject treated of in the paragraph in a very bold black type running on with the text. For each country, forming the subject of a summary, the aforesaid headed paragraphs follow in the same order, and are as follows: To whom granted, for what granted, novelty of invention, duration of patent, date of patent, extent of grant, procedure, official examination, taxes, patents of improvement and certificates of addition, conditions of grant, working invention, etc., etc. By these means a rapid as well as effective comparison of the requirements and advantages in securing patents in foreign countries and at home can be made, and by judicious use of various-sized types and proper spacing, the book can be expeditiously and effectively applied for its purposes without any need of index or table of contents.

**THE GAS AND WATER COMPANIES' DIRECTORY, 1884.** Edited by CHARLES W. HASTINGS (Editor of *The Review of Gas and Water Engineering*, etc., etc.), 22, Buckingham Street, Adelphi, London, W.C. Published by the Scientific Publishing Company Limited, London. Printed by John Smith and Company, Long Acre, 1884.

A WORK of 222 pages, 8vo, bound in cloth. It consists of a directory giving in vertically-arranged columns the town or city referred to, date of formation of gas company, special acts, total share of capital paid up, dividends, total loan capital issued, name of chairman, engineer or manager, secretary, lessee, owner or corporation, population, distance from London, Railway. After giving, as above, details referring to British gas companies and their works, similar details are given referring to Continental and other foreign gas companies. After this follow similarly-arranged details referring to the water companies of this country and abroad. Finally is an alphabetical index of names of managers and engineers, with the towns they represent, giving also the pages in the text which contain the fuller details already above described. Thus if the name of a gas manager or engineer be known, and it be desired to find his location, the latter index gives the reference. If, on the other hand, the locality be known, and the other details required, the text of the work supplies that at once, since the towns and cities are alphabetically arranged opposite the vertical columns which contain all the other information.

**GASWORKS STATISTICS, 1884.** Sixth Issue. Compiled from special returns received from Engineers and Secretaries throughout the United Kingdom, giving (in vertically-arranged columns) tons of coal carbonised, makes of gas and illuminating power, prices per thousand cubic feet, prices paid for public lamps, amounts of dividend, number of consumers, number of public lamps, average prices realised for coke, tons of sulphate made, etc. The towns and cities are alphabetically arranged, and are those of Great Britain and Ireland, and abroad. Edited by CHARLES W. HASTINGS. Published by the Scientific Publishing Company Limited, London.

The work contains 81 pages of matter, it is bound in pasteboard cover, and the price is 3s. 6d.

**WATER WORKS STATISTICS, 1884.** Fourth Issue. Compiled from special returns received from Engineers and Secretaries throughout the United Kingdom, giving source of supply, if gravitation or pumping, quantity raised per annum, if charges based on assessment, if charged by meter, number of meters in use, if constant service, dividend, etc., etc. The towns are

alphabetically arranged, and are those of Great Britain and Ireland, with a few abroad. Arrangements quite similar to that of the Gas Works Statistics. Edited by CHARLES W. HASTINGS. Published by the Scientific Publishing Company Limited, London.

The work contains 29 pages of matter, is bound in pasteboard cover, and its price is 2s. 6d.

**JAHRBERICHT UBER DIE LEISTUNGEN DER CHEMISCHEN TECHNOLOGIE, MIT BESONDERER BERUECKSICHTIGUNG DER GEWERBESTATISTIK FUR DAS JAHR 1883** Von Dr. FERDINAND FISCHER. XXIX. oder Neue Folge XIV. Jahrgang. Mit 471 Abbildungen, Leipzig, Verlag von Otto Wigand, 1884.

THIS work, generally known as "Wagner's Jahresbericht" (R. von Wagner having issued Jahrgänge i.—xxv. of the work) is a thick 8vo volume, bound in paper cover, and containing title page and full table of contents, covering 23 pages. The text itself covers 1,308 pages, and is succeeded by alphabetical indexes of authors and subject matter. The subject matter is divided into groups as follows: Group i., Chemical Metallurgy, covering pages 1—256; ii., Chemical Works Industry (Inorganic Chemistry), pages 257—478; iii., Chemical Works Industry (Organic Chemistry), pages 479—591; iv., Glass, Earthenware, Cement, Mortar and Artificial Stone, pages 592—656; v., Foods, pages 657—1034; vi., Chemical Technology of the Textile Fibres, pages 1035—1135; vii., Other Branches involving Organic Chemistry, pages 1136—1205; viii., Fuel and Electricity, pages 1206—1308. Of course the above division is very distinctly made in the table of contents, and each such division or group receives, moreover, a further subdivision, the nature of which is made plain from the following example taken from group iii.: Chemische Fabrikindustrie, Organisch: Alkohole, 479; Organische Säuren, 481; Cyanverbindungen, 491; Alkaloide n. dgl., 498; Aetherische Oele und Harze, 506; Aromatische Verbindungen, 509; Organische Farbstoffe; (a) Dem Thier- und Pflanzenreiche entstammende Farbstoffe, 535; (b) Theerfarbstoffe; (1) Farbstoffe der Benzolgruppe, 540; (2) Indigofarbstoffe, 560; (3) Pyridin- und Chinolinfarbstoffe, 563; (4) Naphtalinfarbstoffe, 569; (5) Anthracenfarbstoffe, 578; (c) Sonstige organische Farbstoffe und deren Untersuchung, 583.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1881.

5130 S. Gilchrist Thomas, Westminster, and El Biar, Algeria. Improvements in the manufacture of chromic compounds. March 19

5142 W. Weldon, Burstow, Surrey. Improvements in treating the residual liquors of the ammonia soda process, in order to the utilisation of the chlorine of their ammonium chloride. March 19

5153 W. Sharp Mackie, Manchester. An improved process of dyeing india rubber coated fabrics. March 19

5154 J. Inray, London. Manufacture of resin soap. Communicated by H. J. E. Hennebutte, Anglet, France. March 19

5175 J. Brown, Dewsbury. Improvements in disinfecting litter, powder, or compounds. March 20

5182 C. Stuart Gorman, Irvine, Ayrshire. An improvement in the manufacture of bichromate of soda. March 20

5191 W. Travis, London. Improvements in the manufacture of artificial leather. Complete specification. March 20

5192 J. Cowdery Martin, Richmond, Surrey. Improvements in the manufacture of red pigments or paints. March 20

5223 H. Bolz, Braunschweig, Germany. Improvements in the manufacture of non-conducting or insulating material. March 20

5231 J. H. Riley and J. Downham, Bury, Lancashire, and J. Apsley, Rochdale, Lancashire. Improvements in the mode of bleaching linen and jute in the fibres, yarn, and in the woven material manufactured therefrom, and the necessary apparatus used therein. March 21

5237 H. Walley, Manchester, and T. Gare, Stockport, Lancashire. Improvements in protecting from damp, moisture, or decay, and in varnishing and enamelling, and in some cases



hardening or toughening, stone, brick, tile, earthenware, cements, linio, and gypsum, plasters, metals, wood, fibrous materials, paper, yarns, threads, cords, ropes, and woven knitted, and braided fibrous fabrics. March 21

5245 Thomas Slater, Bayswater, Middlesex. Improvements in the manufacture of alloys of chromium and in the utilisation thereof in the coating of metallic surfaces. March 21

5248 A. C. Henderson, London. Improvements in the manufacture of waterproof clothing and linings to articles of wearing apparel. Communicated by Dr. A. B. Boyer, Paris, France. March 21

5262 T. Vosper, London. Improvements in the washing of ores and minerals. March 21

5266 F. W. Nash, London. An improved mode or process of evaporating syrups or other solutions or liquids. Communicated by C. H. Boon, St. Kitts, West Indies. March 21

5267 R. H. Hepburn, Westminster. Improvements in furnaces, and in apparatus for supplying steam and air thereto in order to cause more perfect combustion of fuel. March 21

5269 J. Walker, Leeds, Yorkshire. The manufacture of preparations for antiseptic or preservative purposes and the like. March 21

5276 J. Moulson, Balsall Heath, Worcestershire. A new or improved apparatus for admitting air to steam-boiler and other furnaces for the consumption of the gases arising therein and the prevention of smoke. March 22

5278 Sir William Henry Marling, Bart., King Stanley, Gloucestershire. Improvements in the treatment of wool and other animal products. March 22

5283 E. H. Waldenström and W. Sumner, Manchester. Preventing corrosion of metal tubes. March 22

5317 A. W. Lovell Reddie, London. Improvements in the treatment of vegetable and other tissues or fibres for the purpose of preserving or waterproofing the same. Communicated by MM. Dillies et Cie, Roubaix, France. March 22

5323 J. B. Mackey, London. The preparation of new soluble neutral salts of cerium for medicinal purposes. Complete specification. March 22

5333 L. Lossier, Geneva, Switzerland. An improved process of and apparatus for extracting aluminium from its natural silicates. March 22

5345 C. Billington and J. Newton, Longport, Staffordshire. Alloying manganese iron and tin or lead and manganese iron, tin, and lead. March 21

5357 H. Springmann, Berlin, Prussia. An improved method of utilising the waste materials employed for the purification of lighting gas. Communicated by Dr. Hipp, Hamburg, and Dr. Grünberg, Kalk, Prussia. March 21

5382 H. E. Newton, London. Improvements in explosive compounds. Communicated by A. Nobel, Paris. March 21

5383 H. E. Newton, London. Improvements in the evaporation and concentration of liquids. Communicated by A. Nobel, Paris. March 21

5410 J. Saunders, Liverpool. Improved air pumps and vessels or chambers and devices for exhausting or forcing air or water, and for raising or lowering the temperature, or that of steam, by conduction through the material of which these vessels are composed. March 25

5422 H. H. Lake, London. Improvements in explosive projectiles. Communicated by F. H. Snyder, Jersey, U.S.A. March 25

5427 J. F. Phillips, London. Improvements in apparatus for the extraction of wine. Communicated by A. Rey, Villard-St-Christophe, France. March 25

5430 E. H. Russell, Utah, U.S.A. Improved process of removing metals from ores and metallurgical products, and separating the base from the precious metals. Complete specification. March 25

5431 E. H. Russell, Utah, U.S.A. Improved process of and compound for removing precious metals from ores and metallurgical products. Complete specification. March 25

5445 H. W. Lobb, London. A new method of and apparatus or appliances for eliminating impurities from fermented liquors and alcoholic fluids. March 25

5447 J. Battle Austin, Bristol. Improvements in the manufacture of carbolate of lime for disinfecting and deodorising purposes. March 25

5458 F. Machenhauer, Reddish, Lancashire. Improvements in the manufacture of yellow colouring matters. March 26

5486 W. E. Gedge, London. An improved process for the extraction of fat from cacao, and improved machinery or apparatus in connection therewith. Communicated by Dr. H. Michaelis, Berlin, Prussia. March 26

5488 H. Wedekind, London. Improvements in and connected with kilns for burning bricks or analogous articles. Communicated by H. Dueberg, Berlin, Prussia. March 26

5512 J. H. Johnson, London. Improvements in the preparation of yellow colouring matters suitable for dyeing and printing from tetra-methyl-diamido-benzo-phenone and analogous ketone bases or their halogen derivatives. Communicated by The Badische Aniline and Soda Fabrik, Ludwigshafen-on-the-Rhine, Germany. March 26

5520 W. A. Meadows, Rainhill, Lancashire. Improvements in the manufacture of sulphate of ammonia. March 27

5552 E. Peel Potter and W. H. Higgin, Little Lever, Lancashire. Improvements in the manufacture of bichromate of soda. March 27

5565 B. L. Thomson, London. Improved means for the protection of ships' bottoms and other submerged structures or surfaces from fouling or corrosion. March 27

5595 C. R. Bonne, Manchester. A new or improved yeast-foed, and process for manufacturing the same. March 28

5606 E. H. M. Andreoli, Besançon, France. Converting soft ferrous metals, malleable cast iron, or common steel into the

best class of cemented steel for tools, etc., by rapid distillation of the peat gases. March 28

5628 T. J. Barnard, London. Improvements in the method of and apparatus for economising fuel and utilising the full calorific power of products of combustion. March 28

5668 J. Toussaint, Upper Saitley, Warwickshire. An improvement in the manufacture of steel. March 29

5731 B. J. B. Mills, London. Improvements in the preparation or treatment of wicks for wax and tallow candles and tapers, and for other purposes, in order to prevent smell and smoke on extinction of the same. Communicated by A. Duparquet, Chemin Saint Antoine, Lyons, France. March 31

5741 Improvements in the preparation of yellow, orange, and orange-brown colouring matters suitable for dyeing and printing from tetra-methyl-diamido-benzo-phenone and analogous ketone bases or their halogen derivatives. Communicated by the Badische Aniline and Soda Fabrik, Ludwigshafen-on-Rhine, Germany. March 31

5768 W. Mills, Bodford. Improving the hydraulic or setting properties of limes and cements. Complete specification. April 1

5777 P. M. Justice, London. An improvement in chemical motive powers. Communicated by W. L. Lowrey, Boston, U.S.A. April 1

5794 A. G. Boulton, London. Method of and means or apparatus for generating gas from hydro carbons, and for utilising such gas. Communicated by G. Jones, Washington, U.S.A. Complete specification. April 1

5802 W. R. Lake, London. Improvements in and relating to the manufacture of india rubber and gutta-percha coated fabrics. Communicated by N. S. White, Canton, Massachusetts. Complete specification. April 1

5829 S. Pitt, Sutton, Surrey. Improvements in apparatus for the clarification of liquids. Communicated by W. Oldham, Paris. April 2

5859 E. Padley, Tipton, and W. T. Holt, Willenhall, Staffordshire. Improvements in the means of supplying air to furnaces. April 3

5861 J. Barnett, London. The preservation of perishable substances and the destruction of organisms existing in water. April 3

5873 J. Barnes, Manchester. An improved damp-proof and non-inflammable material for lining walls, covering floors, ships' berths, cabins, and other analogous purposes. April 3

5874 F. Clarke Hills, Deptford, Kent. Improvements in and connected with the treatment of gas-liquor for use in the purification of coal gas. April 3

5887 J. Wetter, London. A manufacture of antimonial compounds suitable as mordants in dyeing and printing. Communicated by M. B. Vogel, Leipzig, Saxony. April 3

5896 A. E. Scott, London. Extraction of metals from carbons or substances known as such, namely, graphite, coal, coke, cinders, and ashes. April 3

5898 W. R. Lake, London. Improvements in the manufacture of ammonia, and apparatus therefor. Communicated by La Société Anonyme Lorraine Industrielle, Hussigny, France. April 3

5923 W. H. Radford, Nottingham. Improvements in the method of utilising the waste heat from direct-acting gas retort furnaces. April 4

5930 T. Terrell, London. Improvements in the destructive distillation of sulphate of iron, and in the obtaining therefrom of sulphurous acid and red oxide of iron. Complete specification. April 4

5932 R. Irvine, Royston Granton, N.B. Improvement in the distillation of coal in the manufacture of coal gas for the subsequent purification of the said gas. April 4

5933 R. Irvine, Royston Granton, N.B. Improvements in the distillation of shale coal or the like for obtaining solid paraffin therefrom. April 4

5936 E. Davies, London. Producing and burning gas or gaseous mixtures, and apparatus therefor. April 4

5945 W. H. Bliss and H. H. Bliss, Ancley, Surrey. Improvements in the manufacture of certain beverages or drinks. April 4

5948 W. J. A. Donald, Glasgow, N.B. Improvements in the manufacture of chromates and bichromates. March 13. (Previously included in No. 481.)

5952 W. Vogel, Chicago, U.S.A. Apparatus and process of consuming smoke. Complete specification. April 4

5963 J. C. Hudson, London. Fireproofing ceilings. April 4

5972 H. Knight, Liverpool. Improvements in pigments containing zinc compounds, and in the manufacture thereof. Complete specification. April 5

5971 J. Lyle, Paisley, Scotland. Improvements in conveying and burning oils for heating, lighting, or consuming smoke. April 5

5975 J. Wilson, Berwick-on-Tweed. Improvements in the manufacture of chlorate of potash. April 5

5984 E. de Pass, London. Improvements in the manufacture of superphosphates. Communicated to him from abroad by E. Dreyfus, Paris. April 5

5996 G. J. Andrews, Harrow Road, and J. E. Sutton, Peckham, Surrey. Improvements in paints or compositions for coating ships' bottoms and other submerged surfaces to prevent fouling, and for preserving iron and wood and other matter from the effect of exposure to salt water, damp, or atmospheric influences. Complete specification. April 5

6004 D. Buros, Carlisle, Cumberland. An improved composition for covering boilers or other vessels or bodies, so as to prevent them losing or gaining heat. April 5

6022 E. F. Trachsel, London. An improved method or process of producing oxide and carbonate of strontium, of barium, or of calcium, and obtaining by-products therefrom. April 5



- 6051 W. V. Wilson, London, and J. Storey, Lancaster. Improvements in the production of compounds containing nitro-cellulose. April 7
- 6082 T. Williamson, Pollockshields, N.B. Improvements in treating or converting iron or making steel, and in furnaces or apparatus therefor. April 8
- 6094 W. Young, Peebles, N.B., and G. T. Beilby, Midcaldor, N.B. Improvements in the distillation of mineral oils, and in the apparatus employed therefor. April 8
- 6116 A. J. Rogers, J. Boebig, and H. Mann, Milwaukee, Wisconsin, U.S.A. Improvements in process and apparatus for reducing metals by electrolysis. April 8
- 6139 A. Gutensohn, London. An improved process for centing the surface of iron or other metal with metallic copper, lead, zinc, aluminium or nickel. April 8
- 6143 A. Myall, London. Improvements in the production of neutral sulphate of alumina by means of magnesia, and in the production of mixtures of sulphate of aluminium and sulphate of magnesium and their application to the sizing of paper. Communicated by F. Lienau, Uerdingen, Germany. April 8
- 6172 G. A. Dick, London. Improvements in the manufacture of metallic alloys. April 9
- 6176 E. Packard, jun., Ipswich, Suffolk. Improvements in obtaining certain solutions containing free phosphoric acid. April 9
- 6179 J. Mangnall, Manchester, and R. S. Lloyd, London. Improvements in the method of generating carbonic acid gas, and in apparatus therefor. April 9
- 6213 W. Wyatt, Ellesmere, Shropshire. Improvements in apparatus for use in the process of softening and purification of water. April 10
- 6215 W. P. Thompson, Liverpool. Improvements in the process of purifying sulphuric acid and of recovering therefrom the arsenic and antimony contained therein. Communicated by G. Thomson, Dillonton, Quebec, Canada, and W. Kemp, Jarrow-on-Tyne, and New Jersey, U.S.A. April 10
- 6229 W. T. Walker, London. Improvements in gas retorts. April 10
- 6231 E. G. Capen and H. Heaton, jun., Kings Norton, Worcestershire. A new or improved compound material for covering steps, stairs, passages, and other things and places where a surface is required which may be walked on safely and noiselessly, and for other like purposes. April 10
- 6259 G. F. Powell, Bristol, Gloucestershire. Improvements in the manufacture of yeast. April 10
- 6271 E. Sonstadt, Cheshunt, Hertfordshire. Improvements in the manufacture of soaps, dentifrices, and other detergent substances. April 12
- 6290 G. Rosenthal, London. Improvements in the manufacture of alumina and of potash alum. April 12
- 6304 E. Sonstadt, Cheshunt, Hertfordshire. Improvements in the preparation of salt, saltpetre, sugar, and other antiseptic substances for use in the preservation of food. April 12
- 6309 G. Halliday, Chelsea. Improvements in sugar mills. April 12
- 6310 J. A. Meylers, Ph.D., Hendon, Middlesex. Improvements in the treatment of locust beans for the purpose of preparing beverages therefrom. April 12
- 6311 C. D. Abel, London. Manufacture of violet colouring matter. Communicated by the Farbwerke vormals: Meister, Lucius, and Bruning, Hoechst-am-Main, Germany. April 12
- 6324 E. G. Brewer, London. A new or improved method of preserving compressible organic substances, especially meat known as preserved corn-meat (creostyptine), fresh, dry, and semi-dried in boxes and packets. Communicated by Dr. G. Compergh, Trieste, Austria. April 12
- 6326 E. Passburgh, Moscow, Russia. A method and apparatus for drying bodies containing quantities of water. Complete specification. April 12
- 6342 J. Woods, Salford, Warwickshire. Improvements in heating kilns, muffles, ovens, and other erections for various purposes, and in bricks, slabs, or blocks to be used therein. April 15
- 6347 P. Ormerod Whitehead, Manchester. Improvements in machinery or apparatus for washing gases or dissolving them in water or other liquids. April 15
- 6351 J. Barratt, Cambuslang, Lanarkshire. Improvements in apparatus to be used in treating or refining vegetable oils. April 15
- 6364 E. A. Brydges, Paris. Improvements in apparatus for raising beer by the generation of carbonic acid gas. Communicated by C. Lauer, Perpignan, France. Complete specification. April 15
- 6367 S. Gilebrist Thomas, London, and El Biar, Algeria. Improvements in the manufacture of sodium. April 15
- 6371 D. G. Fitzgerald, Brixton, Surrey. Improvements in refining gold and silver. April 15
- 6373 J. Cadoux Hudson, London. Improvements in apparatus for the utilisation of petroleum and other combustible fluids as fuel in furnaces. April 15
- 6374 G. Lawrence, London. A new or improved method of and apparatus for producing a flow or circulation of heating liquid between corrugated or similar deflecting surfaces. April 15
- 6376 H. H. Lake, London. Improvements in the manufacture of paper pulp, and in apparatus therefor. Communicated by G. H. Pond, New York, and E. A. Morse, Rutland, Vermont, U.S.A. Complete specification. April 15
- 6383 A. Munzinger, Olten, Switzerland. Improvements in drying apparatus for treating pulp and other substances. April 15
- 6393 C. D. Abel, London. Improved apparatus for brewing purposes. Communicated by C. Zimmer, Frankfort-on-Main, Germany. Complete specification. April 15
- 6402 C. Lowe, Reddish, Lancashire. Improvements in disinfecting, cleansing, and washing compounds. April 16
- 6406 G. Chapman, Glasgow, N.B. Improvements in separating ammonia from blast furnace and other gases and vapours, and in apparatus therefor. Complete specification. April 16
- 6414 L. A. Groth, London. A new or improved process and apparatus for the extraction of metals from alkaline combinations by means of the electrolyte. Communicated by R. Grätzel, Hanover, Germany. April 16
- 6433 H. H. Lake, London. An improved compound metal or alloy, chiefly designed for deoxidising and coating metal plates. Communicated by J. Benbow Jones, Brooklyn, U.S.A. April 16

# THE JOURNAL OF THE Society of Chemical Industry.

## A MONTHLY RECORD

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### NOTICES.

In accordance with the provisions of the 9th by-law, notice is hereby given that those members whose names are placed in italics in the annexed list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Members are hereby invited to nominate fit and proper persons to fill the four vacancies thus created among the Ordinary Members of the Council, in accordance with the terms of the 9th, 11th, and 12th by-laws.

The bye-laws are inserted in the number of the Journal for July, 1883. They can also be obtained from the Secretary, who will supply the prescribed nomination forms upon application.

### ANNUAL GENERAL MEETING.

The next Annual General Meeting of the Members of the Society of Chemical Industry will be held at Newcastle-on-Tyne, on Wednesday, Thursday, and Friday, the 9th, 10th, and 11th of July next.

On the morning of Wednesday, July the 9th, the Members will meet in the Lecture Theatre of the Literary and Philosophical Society, Westgate Road, to transact the ordinary business of the Society, to elect Office Bearers, and to hear an address by the President. In the afternoon they will visit the works of Sir W. G. Armstrong, Mitchell, and Company, at Elswick; and in the evening the Annual Dinner will be held in the Banqueting Hall, Jesmond Dene, by the kind permission of Sir W. G. Armstrong, C.B. Dinner tickets, 10s. 6d. each, exclusive of wine, may be obtained on application, accompanied by Cash or Money Order, to the Honorary Secretary of the Newcastle Section, Mr. J. T. Dunn, 29, Oxford Street, Newcastle-on-Tyne.

A change has been made in the proposed arrangements for the second day of the meeting. It is now arranged that on the morning of Thursday, July the 10th, the Members will proceed by special train to the Lead Works of Messrs. Locke, Blackett, and Company, at St. Anthony's; then to the Alkali Works of the Newcastle Chemical Works Company, at Gateshead; and in the afternoon to the Works of Palmer's Shipbuilding and Iron Company. In the evening a *Conversazione* will be held in the Assembly Rooms, Westgate Road. Tickets for the *Conversazione* will be obtainable at the Reception Room during Wednesday and Thursday.

On Friday, the Members will meet at the Swing Bridge, after inspection of which they will proceed by steamer down the river to Hebburn, where the Alkali Works of Messrs. Charles Tenant and Company, and the Metal Extracting Works of the Tharsis Sulphur and Copper Company, will be visited. After luncheon



in the Drill Hall of 1st Newcastle Volunteer Artillery, the steamer will take the Members across to Willington, to inspect the Lead Works of Messrs. Cookson and Company. The Members will then proceed by the steamer down the river to the pier at at Tynemouth, then returning to Newcastle either by steamer or by train, as they may prefer.

The Wood Memorial Hall of the North of England Mining Institute will be available during the meeting as a Reception and Writing Room. The Library of the Literary and Philosophical Society will also be available as a Writing Room.

Various Works in the district, in addition to those mentioned above, will be open to Members during the Meeting, on presentation of their cards of membership.

Opportunities of seeing in operation the Simon-Carvès process, and the Jameson process, will be afforded during the Meeting.

Messrs. Baird and Company have consented that any Members attending the Meeting who choose to proceed to Gartsherrie for the purpose, shall see the process at work there for obtaining ammonia from the gases from blast furnaces; and Messrs. Young and Beilby have promised to show their process at two Works near Edinburgh.

Full details of the arrangements for the Meeting will be published in the June number of the Journal; and a complete programme of arrangements, with map and plans of Newcastle and district, together with Hotel, Railway, Conveyance, and Postal information, will be furnished to Members at the Reception Room.

An early application for Dinner Tickets is especially desired.

Numerous inquiries have been made for sets of the Journal for 1882. Some of the numbers for that year are out of print. Members who have not the volume for 1882, and wish to obtain it, are invited to signify that wish to the Secretary as early as possible. The numbers out of print are now being reprinted, and will be ready shortly.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 36, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

## PROPOSED AMENDMENT OF BYE-LAWS.

At the forthcoming Annual General Meeting it will be proposed that the Society's Bye-Laws be amended as follows:—

1. That Clause (b) of Bye-Law 2 be omitted.
2. That Clause (c) of Bye-Law 2 shall become Clause (b), and shall be modified so as to read as follows: "To

afford its Members opportunities for the interchange of ideas with respect to improvements in the various chemical industries, and for the discussion of all matters bearing upon the practice of applied chemistry, and to publish information thereupon."

3. That Clause (d) of Bye-Law 2 shall become Clause (c), and that the word "purpose" therein shall be changed to "purposes."

4. That Clause (e) of Bye-Law 2 shall become Clause (d).

5. That all of Bye-Law 3 which follows the word "Sections" in the third line of that Bye-Law shall be omitted.

6. That Bye-Law 5 shall be omitted.

7. That Bye-Law 6 shall become Bye-Law 5.

8. That Bye-Law 7 shall become Bye-Law 6, and that for the portion of it following the word "in" the following shall be substituted: "such manner as the Council may think fit for the purposes of the Society as defined by its Bye-Laws."

9. That for Bye-Laws 8 to 15 the following shall be substituted:—

(7) Except as otherwise provided by Bye-Laws 4, 23, and 24, all office bearers shall be elected at Annual General Meetings of the Society, and by ballot.

(8) The persons to be submitted for election at Annual General Meetings to the offices of President, Vice-President, Honorary Treasurer, and Honorary Foreign Secretary, shall be nominated by the Council. Those to be submitted for election to the office of Ordinary Member of Council shall be nominated as provided by Bye-Laws 17, 18, and 19.

(9) The Honorary Treasurer and the Honorary Foreign Secretary shall retire annually, but shall be eligible for re-election.

(10) The President shall hold office for one year, retiring at the close of the Annual General Meeting next following that at which he was elected, but shall be eligible for re-election.

(11) When the President who is about to retire is not nominated for re-election to the office of President, he shall always be one of the persons nominated by the Council for election to the office of Vice-President.

(12) Four Vice-Presidents shall retire each year, and those who thus retire shall not be eligible for re-election to the Council, except under Bye-Laws 23 and 24, until the Annual General Meeting next following that at which they retire. When a Vice-President is nominated by the Council for election to the office of President, the Vice-President thus nominated shall not be counted among the four Vice-Presidents who are to retire.

(13) Except that any Vice-President may be nominated by the Council for election to the office of President, and that such Vice-President, if any, shall not be included in the operation of the present Bye-Law, the Vice-Presidents shall retire as far as may be possible in the order in which they were elected. When the four who must retire cannot all be Vice-Presidents who have been in office for the longest period, such number of them as may be necessary shall be selected by the Council by ballot from among those of the Vice-Presidents who have been in office for the next longest period.

(14) Any Member of the Society who is not in arrear with his subscriptions except as otherwise provided by Bye-Law 12, may be nominated and elected to the office of Vice-President.

(15) Four Ordinary Members of Council shall retire each year, and those who thus retire shall not be eligible for re-election to the Council, except under Bye-Laws 23 and 24, until the Annual General Meeting next following that at which they retire. When an Ordinary Member of Council is nominated for election to the office of President, and when an Ordinary Member or two or more Ordinary Members of Council may have been nominated for election to the office of Vice-President, the Member or Members so nominated shall not be counted among those who are to retire.

(16) Except that any Ordinary Member of Council may be nominated for election to the Office of President, or to that of Vice-President, and that the Member or Members, if any, who have been so nominated, shall not be included in the operation of the present Bye-Law, the Ordinary Members of Council shall retire as far as may be possible in the order in which they were elected. When the four who must retire cannot all be Members who have been in office for the longest period, such number of them as may be necessary shall be selected by the Council by ballot from among those of the Ordinary Members of Council who have been in office for the next longest period.

(17) If, by reason of any Ordinary Member of Council, or any two or more Ordinary Members of Council, having been nominated for election to the office of President or to that of Vice-President, the number of Ordinary Members of Council to be elected at the ensuing Annual General Meeting shall be greater than four, the Council shall nominate the number in excess of four of the persons to be submitted to that Annual General Meeting for election as Ordinary Members of Council, and four shall be nominated as provided by Bye-Laws 18 and 19.

(18) At least two months before the date of each Annual General Meeting, the Council shall issue to each Member of the Society a list of the Council, showing which of its Members are to retire at the date of that Meeting, and giving the names of the persons who have been nominated by the Council for election at that Meeting to the office of Vice-President, and also the names of the persons, if any, who have been nominated by the Council under Bye-Law 17 for election to the office of Ordinary Member of Council, and inviting nominations to the four of the vacancies about to occur, persons to fill which have not been nominated by the Council. Such nominations must be made upon a printed form, which will be supplied to any Member of the Society on his request addressed to the General Secretary, and which form shall be as follows:—

[FORM.]

No such nomination shall be valid unless it be signed by at least ten Members of the Society, who are not in arrear with their subscriptions, nor unless it be received by the General Secretary, at the Society's office, at least one month before the date of the commencement of the Annual General Meeting to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Bye-Law 12 or Bye-Law 15.

(19) If the number of persons nominated as provided by Bye-Law 18 be fewer than four, the further number necessary shall be nominated by the Council. If the number of persons nominated as provided by Bye-Law 18 be in excess of four, the Council shall select the four having the largest number of nominators.

(20) A complete list of the Council, showing which of its Members will retire at the forthcoming Annual General Meeting, and the persons who have been nominated for election at that Meeting, shall be printed as a Ballot List, and this Ballot List shall be sent to each Member of the Society at least five days before the commencement of the said Meeting.

(21) On the first day of the said Meeting, at such stage of its proceedings as shall be most convenient, the Members present shall hand their Ballot Lists to the General Secretary, or, in his absence, to such other person as the President may appoint. Members unable to attend the Meeting may send their Ballot Lists before the Meeting to the General Secretary, in sealed envelopes. Each such sealed envelope must be marked **BALLOT LIST**, and must moreover have written upon it the name of the Member sending it. The names written upon such sealed envelopes having been compared with the Society's Register by the General Secretary, such sealed envelopes shall be opened at the Meeting by

Scrutators appointed by the Meeting. The Scrutators shall see that the conditions of the ballot are strictly fulfilled.

(22) Any Member shall be at liberty to strike out any name or names printed on his ballot-list, and to substitute for the name or names which he strikes out the name or names of any other Member or Members of the Society who are not ineligible for election by Bye-Law 12 or Bye-Law 15; but he must not write upon his ballot-list more new names than the number of printed names which he strikes out.

(23) If the office of President should become vacant by death or resignation during the period intervening between two Annual General Meetings, a new President shall be elected by the Council.

(24) If any other vacancy shall occur in the Council during the period intervening between two Annual General Meetings, the Council shall elect a person to fill it until the next Annual General Meeting, when the Member who has been so elected by the Council shall retire in addition to those who retire under Bye-Laws 12 and 15. Such Member, however, shall be eligible for re-election, and shall be nominated for re-election at the Annual General Meeting next following his election by the Council.

10. That Bye-Laws 16, 17, 18, and 19, shall become respectively Bye-Laws 25, 26, 27, and 28, and that for the words following the word "Society" in Bye-Law 16, the following shall be substituted: "At whatever period of the year a new Member be elected, he shall be required, unless the Council shall determine otherwise, to pay his subscription for that year; having done which, he shall be entitled to receive all the numbers of the Society's Journal for that year."

11. That Bye-Law 20 shall become Bye-Law 29, and that the portion of it following the word "subscription" in its second line, and ending with the word "liability" in its seventh line, shall be deleted.

12. That Bye-Law 21 shall become Bye-Law 30, and that for the portion of it following the word "subscription" in its second line the following shall be substituted: "Any Member may withdraw from the Society at the end of any year, after giving notice to the General Secretary of his desire to withdraw, such notice to be accompanied by the payment of all arrears, if any, which may be due from him to the Society up to that date, and by such notice and payment he shall be released from all further liability as a Member."

13. That the following shall be Bye-Law 31: "The Council shall meet periodically, to examine accounts, to authorise payments, to act as a Committee of Finance, and to transact such business as may be necessary. Its meetings shall be convened by circular posted to all its Members, either by the order of the Council itself, given at a previous meeting, or by instruction from the President; and at any meeting of the Council thus duly convened five Members shall constitute a quorum. The circular convening each meeting shall be accompanied by an agenda paper, stating the nature of the business to transact which the meeting is called, and also by a list of the candidates for membership, if any, who are to be proposed for election at such meeting, with the address and occupation of each such candidate."

14. That Bye-Law 25 shall become Bye-Law 33. That for the words "or processes in chemical engineering or manufacturing matters" therein shall be substituted the words "processes, or other matters of interest in connection with the chemical industries," and that the final sentence of that Bye-Law shall be deleted.

15. That the following shall be Bye-Law 34: "No Special Committee, nor any Committee of a Local Section, shall have any control over the funds of the Society, except to the extent to which funds for its use may be voted by the Council. An account of the manner in which any funds so voted for the use of any Committee have been expended shall be furnished to the Council on or before the 31st of December in every year."

16. That Bye-Law 22 shall become Bye-Law 35, and that the latter portion of it shall read as follows: "to receive a Report from the Council, to elect office bearers,



to hear an Address by the President, and to transact such other business as may be necessary or desirable, including, if the Council think fit, the reading and discussion of papers."

17. That Bye-Law 23 shall become Bye-Law 36, and shall read: "Notice of each Annual General Meeting shall be given in the Journal of the Society at least fourteen days before the day on which such meeting is to commence."

18. That the following shall be substituted for Bye-Law 28, and shall be Bye-Law 37: "The order of the business to be transacted at the Annual General Meetings shall be decided by the Council from time to time."

19. That Bye-Laws 29 and 30 shall become Bye-Laws 38 and 39.

20. That Bye-Law 31 shall become Bye-Law 40, and shall read: "If any person proposed to the Council for election as a Member of the Society be rejected, no record of his rejection shall be placed upon the minutes."

21. That Bye-Law 32 shall be deleted.

22. That Bye-Laws 33 and 34 shall become respectively Bye-Laws 41 and 42.

23. That Bye-Law 35 shall become Bye-Law 43, and that for the words following the word "Society" in it the following shall be substituted: "except that no Member of the Society shall be entitled to receive it who is in arrear with his subscription."

24. That Bye-Law 36 shall become Bye-Law 44, and shall read as follows: "The author of any communication which has been read before the Society or before any of its Local Sections, and the publication of which in the Society's Journal has been authorised by the Council, may receive not more than 50 copies of such communication, together with the discussion thereon, if the author desires it, printed separately, provided that he gives notice of his desire to receive such copies upon the manuscript of his communication forwarded by him to the Editor of the Journal."

25. That Bye-Law 37 shall become Bye-Law 45, and shall be modified as follows: "The Journal of the Society shall be edited and managed as the Council shall prescribe from time to time. Nothing shall appear in it the publication of which has not been authorised by the Council, or by a Publication Committee appointed by the Council. It shall contain, in full or in abstract, as the Council may in each case decide, such of the communications read before the Society or any of its Local Sections as the Council may consider it desirable to publish, together with such other matter, relating to applied chemistry and the practice thereof, as the Council may from time to time direct."

26. That the present Bye-law 26 shall be deleted, and that Bye-law 38 shall become Bye-Law 46, and conclude with the word "formed" at the end of its first sentence.

27. That the following shall be substituted for Bye-Law 39, and shall be Bye-Law 47: "Each Local Section may make rules for its own government, but no such rules shall be valid unless and until they have been sanctioned by the Council of the Society, and such rules must in each case include the following provisions:

"(a) That the affairs of the Section shall be conducted by a Committee, which shall be elected by the Members of the Section, in such manner as the rules of the Section shall provide.

"(b) That, except elections to fill up unforeseen vacancies, all elections to the Committee of a Local Section, and all election of officers of the Section, whether such officers be appointed by the Committee or otherwise, shall take place in the month of April, and that the names of the persons elected shall in each case be reported to the Council on or before the 20th of that month; but that the members of Committee and the officers thus elected or appointed in April shall not take office, and those whom they are to succeed shall not retire from office, in each case until the close of the session then current."

"(c) That no modification or addition to the rules of the Section shall be made without the consent of the Council of the Society."

28. That the following shall be Bye-Law 48: "When the Council has consented to the formation of a Local Section of the Society, and has approved the rules by which it is proposed that that Section shall be governed, the Chairman and Honorary Secretary of that Section shall be *ex-officio* Members of the Council of the Society."

29. That the following shall be substituted for Bye-Law 40, and shall be Bye-Law 49: "Each Local Section shall be bound to defray its own expenses for printing, stationery, postage, reporting, and hire of rooms; but the Council shall make a grant in aid thereof out of the general funds of the Society, or may, if it think fit, defray the whole thereof."

30. That the following shall be substituted for Bye-Law 41, and shall be Bye-Law 50: "An account of the expenditure of each Local Section during the preceding twelve months shall be furnished to the Council on or before the 31st of December in each year."

31. That the following shall be substituted for Bye-Law 46, and shall be Bye-Law 51: "Except for printing, stationery, reporting, postage, and hire of rooms, no Local Section shall incur any expense without having previously obtained the sanction of the Council thereto."

32. That the following shall be substituted for Bye-Law 42, and shall be Bye-Law 52: "For the purpose of meeting its expenses, each Local Section may require its Members to pay a Sectional subscription, which, however, shall not in any case exceed five shillings per annum."

33. That Bye-Law 43 be deleted.

34. That the following shall be substituted for Bye-Law 44, and shall be Bye-Law 53: "Every Member of the Society shall be entitled to attend, and to take part in the proceedings of, the meetings of all Local Sections, except meetings concerning the business management of any Section of which he is not a Member."

35. That the following shall be substituted for Bye-Law 45, and shall be Bye-Law 54: "The Committees of Local Sections shall have power to accept or reject communications proposed to be read before such Sections."

36. That the following shall be substituted for Bye-Law 47, and shall be Bye-Law 55: "Any Member of the Society may submit to the Council in writing any proposal to alter or add to the present Bye-Laws, and the Council may submit such proposal to the ensuing Annual General Meeting, if it think fit; but the Council shall submit such proposal to such Annual General Meeting on a requisition to that effect signed by at least twenty Members of the Society, who are not in arrear with their subscriptions, provided that no proposed alterations or additions to the Bye-Laws shall be considered at any Annual General Meeting unless at least one month's notice thereof has been given to every Member of the Society."

37. That Bye-Law 27 shall become Bye-Law 55.

#### LIST OF MEMBERS ELECTED MAY 23rd, 1884.

W. Mathwin Angus, St. John's Leather Works, Newcastle-on-Tyne.

Dr. C. G. Auerbach, Farbenfabriken vorm. F. Bayer & Co., Elberfeld.

E. Barlow, 2, Railway Terrace, Streatham, S.W., compressed gas maker.

J. J. Beringer, A.R.S.M., 10, South Terrace, Camborne, Cornwall, lecturer.

Gustav Bischof, 4, Hart Street, Bloomsbury, W.C., anal. and tech. chem.

Wm. Black, Stanrigg, Airdrie, N.B., coal master.

R. Bowman, Heyworth Alkali Works, Gateshead-on-Tyne, analyst.

Pythagoras Boyd, Box 77, North Adams, Mass., U.S.A., printworks chemist.

W. Buckley, Brookside Printworks, West Leigh, Lancashire.

L. N. Chadwick, Ivy Lawn, Ponder's End, Middlesex, manager, crape works.

L. Cropper, Eagley Mills, near Bolton, manager.

A. de Deken, Braddock, Pa., U.S.A., analyst.

R. Dempster, Rose Mount, Eland, Yorkshire, gas engineer.

C. S. Doggett, Boston, Mass., U.S.A., and Polytechnicum, Zurich, student.

Wm. Foulis, 2, Montgomerie Quadrant, Kelvinside, Glasgow, gas engineer.

W. A. Fox, Court, Wellington, Somerset, woollen manufacturer.

Joe Frost, Moldgreen, Huddersfield, manufacturing chemist and analyst.  
 J. Gaskell, 1, Woodlands Road, Cheetham Hill, Manchester, analyst.  
 J. Handcock, Dimsdale View, Longport, Staffordshire, potter's manager.  
 Chas. Heap, Caldershaw, near Rochdale, finisher, dyer, and fuller.  
 Wm. Holloway, Newlands, Middlesbro', manufacturing chemist.  
 J. W. Ingham, 2, Bisley Villas, Ferndale Road, Leytonstone, E., chemist.  
 J. Jameson, Akenside Hill, Newcastle-on-Tyne, consulting engineer.  
 Dr. Wm. Kalle, Biebrich, am Rhein, Germany, tar colour manufacturer.  
 T. J. King, Inland Revenue Laboratory, Somerset House, W.C., analyst.  
 J. Kolb, Société Anonyme des Manufactures de Prod. Chim., Lille, France.  
 H. W. Lake, States Analyst's Office, Jersey.  
 Baldwin Latham, M. Inst. C. E., 7, Westminster Chambers, Victoria Street, S.W.  
 L. Liebermann, 52, Portland Street, Manchester, agent for Messrs. Meister, Lucius, und Brueening.  
 A. S. Lodge, Spring Side, Prestwick, near Manchester, science student.  
 James Lyle, Plaistow Wharf, North Woolwich Road, E., sugar refiner.  
 Edward Marsden, Church View, North Road, Clayton, Manchester, dyer, printer, etc.  
 Dr. E. von Salis Mayenfeld, 6, North Road, Clayton, Manchester, Chemist Aniline Co.  
 J. McCulloch, 2, Tyne View Terrace, Hebburn, assistant chemical manager.  
 B. T. Moore, Cavendish House, Teddington, Middlesex, M. Inst., C.E.  
 J. B. Moorhouse, Horton Bank, Bradford, dyer.  
 H. S. Murray, Glenmayne, Galashiels, N.B., tanner.  
 A. H. Newton, Belsize Court, Hampstead, N.W., colour maker.  
 Saml. Pickard, Springfield Paper Mills, Bolton, paper maker.  
 Colin Rafter, 9, Valentine Street, Pendleton, Manchester, manufacturer.  
 Edwin Redfern, 17, Clayton Street, Hulme, Manchester, cashier.  
 Chas. Renard, 43, Rue Grignan, Marseilles, France, "Rio Tinto Co."  
 Wm. B. Ritchie, The Grove, Belfast, chemical manufacturer.  
 R. Sandon, 118, Leighton Road, Kentish Town, N.W., examiner, Patent Office.  
 C. Schroeder, 55, Bloom Street, Manchester, drysalter.  
 Jas. Smiles, 3, Brandon Terrace, Edinburgh, manufacturing chemist.  
 H. W. Southcombe, Fairview, Yeovil, leather manufacturer.  
 W. G. Thompson, 5, Cooper Street, Manchester, aniline dye maker.  
 G. D. Thudichum, 11, Pembroke Gardens, W., analyst.  
 J. H. Tibbitts, Astoria, Long Island, New York, U.S.A., manufacturing chemist.  
 Geo. Tunbridge, Howard Villa, Belgrave Road, Leicester, gas manager.  
 T. Venables, 4, Holborn Terrace, Kelvinside, Glasgow, chemical manufacturer.  
 S. R. Walker, 21, School Street, Radcliffe, Manchester, form-dyer.  
 C. S. Stanford Webster, Malvern House, Redland, Bristol, professor.  
 F. Whowell, Carr Bank, Tottington, Bury, bleacher and finisher.  
 W. Watson Will, Ossory Villa, Ossory Road, S.E., professor of organic chemistry.  
 C. Greville Williams, F.R.S., St. Stephen's Road, Hounslow.  
 Henry Bassett, 26, Belitha Villas, Barnsbury, N.

#### CHANGES OF ADDRESS, ETC.

Barks Fred, (late Canterbury College), New Zealand Loan and Mercantile Agency Co., Christchurch, New Zealand.  
 Bishop, G. A. (late of Hamilton, N.B.), Royal Bank House, Coatbridge, N.B.  
 Bishop, Howard W. (late of Walsall), Wigginton Road, Tamworth.  
 Bower, H., P.O. Box 946, Philadelphia, Pa., U.S.A.  
 Carruthers, Robert (late of Glasgow), Pow Street, Workington, Cumberland.  
 Gilchrist, Peter S. (late of Salterhebble), 10, Burlington Crescent, Goolse.  
 Greenway, T. J. (late of Cordoba), 55, Digby Road, Brownwood Park, N.  
 Hall, W. J. (late of Wappinger's Falls), New Jersey Extraction Works, New Jersey, Elizabeth, U.S.A.  
 Hampson, A. (late of Gorton), Leven Terrace, Fairfield, near Manchester.  
 Higgin, A. J., 22, Little Peter Street, Gaythorn, Manchester.  
 Hutchinson, T. J. (late of Elton), Leeman's Hill Works, Tottington, near Bury.  
 Johnstone, Jas. (late of Paisley Road), Shawfield Works, Rutherglen, Glasgow.  
 Knight, J. J. (late of Greenheys), Aluminium Crown Metal Works, Solihull Lodge, Holyrood, near Birmingham.

Martyn, Wm. (late of Elizabeth), care of The Davis Co., 3 Merchant's Exchange, Boston, Mass., U.S.A.  
 Reed, Albert E., 21, St. Andrew's Crescent, and Ely Paper Mills, Cardiff.  
 Richardson, B. S. (late of Consett), Scottsdale, Westmoreland Co., Pa., U.S.A.  
 Richardson, F. W. (late of Church Street), 13, Whetley Grove, Bradford.  
 Robinson, Jos. (late of Irwell Street), Farnworth, Widnes.  
 Rohling, Ferd. (late of Manchester), Overlea Terrace, Sheddens, Busby, near Glasgow.  
 Sanson, A. (late of Castellamare), 25, Hullard Street, Shrewsbury Street, Old Trafford, Manchester.  
 Shapleigh, Waldron (late of Freeport), care of Graham Blandy, Esq., 42, Broadway, New York.  
 Smith, George, Ravensdene, Sutton, Surrey, and Anchor Bottling Works, Bow Bridge, E.  
 Toms, F. Woodland (late of Camden Town), States Analyst's Office, St. Heliers, Jersey.  
 Warne, Thos. (late of South Shields), 22, Foley Street, Wednesbury.

#### CHANGE OF ADDRESS REQUIRED.

Seckendorf, A. (late of 17, Ashley Place), Victoria Street S.W.

### Deaths.

DR. R. ANGUS SMITH, F.R.S., 22, Devonshire Street, All Saints, Manchester.

THOMAS FARMER, West Gorton, Manchester.

G. M. HOPWOOD, Melbourne, Australia.

PROF. A. F. TAYLOR, Cleveland, Ohio, U.S.A.

### London Section.

Chairman: David Howard.

Committee:

Sir F. A. Abel.	B. E. R. Newlands.
H. E. Armstrong.	B. Redwood.
W. Crowder.	T. Royle.
C. Graham.	John Spiller.
S. Hall.	G. C. Trewhby.
A. K. Huntington.	W. Weldon.
R. Messel.	J. Williams.

Local Sec.: Thos. Tyrer, Garden Wharf, Church Road, Battersea, W.

On June 9th, the following papers will be presented by—

1.—Mr. W. S. Squire, "On the Processes concerned in the conversion of Starch into Alcohol, and their relation to Brewing and Distilling."

2.—Mr. H. B. Yardley, "On Iron and Alumina as causes of reduction in the manufacture of Superphosphates, &c."

3.—Messrs. Giles and Shearer, "On Table of the Percentage of Sulphurous Acid by weight in its Aqueous Solutions."

4.—Messrs. Pedler and Warden, "On the Effect of Temperature on the Flashing Point of Petroleum."

CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE

Monday, May 5th, 1884.

MR. DAVID HOWARD IN THE CHAIR.

### THE COMPOSITION AND ILLUMINATING POWER OF COAL GAS.

BY PERCY F. FRANKLAND, PH.D., B.Sc., F.I.C.,  
 ASSOC. ROY. SCH. OF MINES.

THE composition of coal gas is a subject which has attracted the attention of chemists for some 50 years past, and although it has undergone investigation by men of great eminence, yet our knowledge of it still remains far from perfect. Indeed coal gas possesses such



a complex composition, and most of its ingredients are present in such small quantities only, that the ordinary methods of analysis are inadequate to deal with them. Even the analysis of far simpler gaseous mixtures is an operation which is by no means popular, owing to the great amount of care, special skill, and expensive apparatus which are requisite. It is not, therefore, surprising that the published analyses of coal gas should be comparatively limited in number.

Although by searching for individual constituents in very large quantities of gas upwards of 20 different ingredients of coal gas are reported to have been discovered, yet the quantitative determination of most of them is altogether out of the question. For practical purposes it is necessary to be content with a much less exhaustive analysis of coal gas, and yet one which shall discover the connection between the composition and illuminating power of the gas. These numerous ingredients of coal-gas may be conveniently divided into three groups:—

1. *Illuminating Constituents*, which consist chiefly of ethylene,  $C_2H_4$ , propylene,  $C_3H_6$ , butylene,  $C_4H_8$ , acetylene,  $C_2H_2$ , and benzene,  $C_6H_6$ .

2. *Diluents*, which are represented by hydrogen, marsh-gas, and carbonic oxide.

3. *Impurities*, the principal of which are carbonic anhydride, nitrogen, atmospheric air, and aqueous vapour.

Now although an analysis in which the proportion of illuminating constituents, as well as the hydrogen, carbonic oxide, marsh-gas, carbonic anhydride, nitrogen, and oxygen are determined, can be performed in a few hours, yet such analyses are but rarely made, the testing of gas being generally confined to the determination of its illuminating power and an estima-

tion of certain impurities, generally sulphur and ammonia, only. It is needless, however, to point out the importance of such analyses, not only in order to ascertain the calorific effect, the amount of carbonic anhydride produced, or the oxygen consumed in the combustion of the gas, but even as regards its illuminating power, these analytical results indicate more clearly the amount of light that the gas will yield to an ordinary consumer than the official photometric determinations made with elaborate standard burners which are never seen in private houses.

The great importance of determining the composition of the gas resulting from different methods of gas manufacture, is also sufficiently obvious.

The composition of the gas supplied to London was fully examined in the year 1851, and again in 1876, the results of these analyses are given in the accompanying tables. In these analyses, it will be seen that not only have the principal constituents been determined, but the nature of those hydrocarbons to which the illuminating power of the gas is due has been further explored. The illuminating power of the hydrocarbons of the general molecular formula  $C_nH_m$  is known to increase as the value of  $n$  increases, and that of  $m$  diminishes. Thus not only does propylene,  $C_3H_6$ , yield more light, on combustion, than ethylene,  $C_2H_4$ , but the latter is inferior also to acetylene,  $C_2H_2$ . In order, therefore, to form an opinion concerning the illuminating value of these hydrocarbons  $C_nH_m$  their "carbon density," i.e., the number of carbon atoms which they contain in a molecule, was also determined. This value of the hydrocarbons may conveniently be expressed in terms of ethylene, the carbon density of which is 2.

TABLE I.—1851 (E. FRANKLAND).

100 volumes of Gas contain

	COMPANY.	Carbonic Acid.	Oxygen.	Nitrogen.	Hydrogen.	Carbonic Oxide.	Marsh Gas.	Heavy Hydro- carbons.	Carbon Density.	Equivalent in Olefant Gas.
1	City Co., Taken at Blackfriars (Newcastle Coal).	·53	..	..	17·60	7·32	41·50	3·05	1·57	6·97
2	Great Central Co., Taken at Office, Cole- man Street, 3 miles from Works (New- castle Coal and Can- nel).	·28	·44	1·80	51·21	7·10	35·28	3·56	1·05	7·21
3	Western Co., Taken at Works, Pad- dington (Newcastle Cannel).	·13	·13	1·51	25·82	7·85	51·20	13·06	3·52	22·08
4	Imperial, Taken $\frac{1}{2}$ mile from Battle Bridge Works.	·29	1·20	5·01	41·15	8·02	40·66	3·67	3·35	6·15
5	Chartered, Taken $\frac{1}{2}$ mile from Works, Brick Lane.	None	·08	·38	51·81	8·95	35·25	3·53	1·36	7·70

These analyses brought to light the interesting fact that during the interval of 25 years which elapsed between the two series of observations, the illuminating power of the gas, as indicated by its chemical composition, had undergone no increase, the improved illuminating power which had nominally taken place being really due to the superior burners which had come into use for testing the gas.

These analyses also show the difference in composition between the gas prepared from common coal,

cannel coal, and mixtures of the two respectively. It will also be seen that whereas carbonic anhydride forms an almost constant ingredient of the gas in 1851, it had been practically banished by the year 1876. Nitrogen, on the other hand, becomes a more conspicuous ingredient in the later analyses.

During the past two years I have myself been investigating the composition not only of the gas supplied to London by the two principal companies, but also of that supplied to a number of the more

TABLE II.—1876 (HUMPIDGE).  
 100 volumes of gas contain

	COMPANY.	Carbonic Acid.	Oxygen.	Nitrogen.	Hydrogen.	Carbonic Oxide.	Marsh Gas.	Heavy Hydrocarbons.	Carbon Density.	Equivalent in Olefant Gas.
1	Imperial Co., Crane Grove, Holloway Road, 10.30 p.m.	None	1.91	9.73	10.82	6.79	36.57	4.18	3.81	7.96
2	Chartered Co., Beckton Testing House, Noon.	None	Traces	3.18	50.59	3.13	38.39	4.11	3.56	7.85
3	Chartered Co., Mr. Heisch's Office, 11.30 a.m.	None	0.22	4.98	50.28	4.37	36.95	3.22	3.94	6.31
4	Ditto 9.30 p.m.	None	0.60	3.22	50.62	6.96	34.49	4.11	3.16	6.49
5	Chartered Co., Royal College of Chemistry, Noon.	Traces	0.26	4.93	50.68	3.98	35.89	4.08	3.26	6.65
6	Ditto 9 p.m.	Traces	0.40	5.40	43.99	6.42	39.36	4.12	3.31	6.82
7	Houses of Parliament (Cannel), 7 p.m.	None	None	2.71	41.72	4.98	41.88	8.72	1.51	19.66

 TABLE III.  
 COMPOSITION OF COAL AND CANNEL GASES (P. F. FRANKLAND), 1882-4.

TOWN.	Reputed Illuminating Power in Standard Candles.	Hydro-Carbons, Cn Hm.	Equivalent in Ethylene, C <sub>2</sub> H <sub>4</sub> .	Average Formula of Cn Hm, or		Carbonic Anhydride, CO <sub>2</sub> .	Oxygen.	Nitrogen.	Hydrogen.	Carbonic Oxide CO.	Marsh-Gas, CH <sub>4</sub> .	Ratio Illuminating Power. Equivalent of C <sub>2</sub> H <sub>4</sub> or 1% C <sub>2</sub> H <sub>4</sub> = Stand. Candles.		Price per 1,000 Cubic Feet.
				Carbon Density.	Hydrogen Density.									
Edinburgh.....	(302)	12.23	16.55	2.71	5.38	.35	1.00	3.61	33.21	6.61	12.93	1.81		3.10
Glasgow .....	27	10.00	13.40	2.68	5.00	.29	.06	3.07	39.18	7.14	10.26	2.01		3/8
St. Andrews.....	27	10.04	13.71	2.73	..	2.73	.18	2.83	36.63	5.16	12.13	1.97		1/2
Liverpool.....	21.5	7.90	9.50	2.11	..	1.70	.19	6.10	36.41	3.39	14.28	2.26		2/10
Preston .....	20	6.22	8.41	2.70	..	.84	.25	4.79	43.95	4.62	39.33	2.38		3.0
Nottingham.....	18.5	5.63	8.24	2.93	..	.31	.24	2.51	45.52	5.63	39.66	2.25		2.6-2/8
Leeds.....	18	7.28	10.64	2.92	..	.31	.07	1.32	40.23	5.02	42.74	1.69		1/10
Sheffield .....	18	6.28	8.78	2.79	..	.24	.10	2.56	43.05	4.72	43.05	2.05		2.0-2/4
Birmingham.....	17.25	4.76	6.28	2.61	4.29	1.50	.36	10.10	40.23	4.05	39.00	2.75		2.3-2.9
Bristol .....	17	4.58	7.77	3.39	..	0	.27	5.11	41.57	4.77	40.70	2.19		2.8-2/10
London .....	..	..	..	..	..	..	..	..	..	..	..	..		..
Gas Light & Coke Co. ....	16	4.41	6.58	2.98	4.32	0	.26	5.95	47.99	3.75	37.64	2.43		3.2
South Metropolitan Co. ....	16	2.92	4.42	3.03	5.16	.09	0	3.19	53.44	4.11	36.55	3.62		2.10
Redhill .....	16	4.10	5.91	2.69	4.58	.74	.49	3.37	48.18	3.41	39.41	2.71		4/6-5/-
Gloucester .....	(162)	4.95	7.10	2.87	..	.03	.51	2.73	48.89	4.61	38.25	2.25		..
Newcastle-on-Tyne..	16	3.62	5.00	2.76	..	.28	.23	5.29	50.50	3.37	36.71	3.20		1/10½
Newcastle-under-Lyme.....	15	4.53	5.67	2.47	..	.08	.11	6.22	46.31	3.74	39.01	2.65		3.6
Brighton.....	14	3.76	4.60	2.15	4.62	.03	.23	2.07	51.62	4.14	38.15	3.04		3.3-5.3
Southampton.....	11	3.09	4.90	3.17	5.20	.07	.39	2.53	53.59	3.59	36.71	2.86		3.2-3.8
Ipswich.....	14	4.53	5.82	2.57	3.77	.06	.12	10.84	43.26	2.46	38.73	2.40		3.6
Norwich .....	(112)	3.26	4.85	2.97	3.40	.27	.14	3.03	53.79	3.10	36.11	2.89		2.6-2.8



important towns in England and Scotland. The results of these analyses are embodied in the accompanying table. I have also appended the reputed illuminating power of the gases in question.

The results are instructive, as they show the limits within which the ingredients of ordinary coal-gas vary. They also show that although the composition of the coal-gas manufactured in this country to-day is much the same as it was 33 years ago, yet there are some minor differences which are not without interest.

Referring in the first place to the illuminating hydrocarbons, it will be seen that although the proportion in which they are present has generally increased yet their carbon density is noticeably lower. Already in 1876 this carbon density was generally inferior to that observed in 1851. More especially is this difference apparent in the case of cannel-gas, thus in 1851 the Metropolitan cannel-gas of the Western Co. contained hydrocarbons of the average carbon density 4.05, in 1876 similar gas supplied to the Houses of Parliament contained hydrocarbons of the density 4.51, whilst the various cannel-gases which I have examined, viz., from Liverpool, Glasgow, Edinburgh, St. Andrew's, and others, only contain hydrocarbons the average density of which is considerably under 3. There can be little doubt that this reduction in the carbon density of the hydrocarbons is due to different conditions of temperature which are now employed in destructive distillation.

In some cases, it will be seen, I have also determined the hydrogen density of the hydrocarbons, which determination throws light upon the family of hydrocarbons which is most predominant. Thus from the carbon density alone it would be impossible to distinguish between  $C_2H_4$  and  $C_2H_2$ , whilst the hydrogen density at once serves to discriminate between them. The determinations of carbon and hydrogen density show that the illuminating power of the cannel-gases is due almost wholly to hydrocarbons of the  $C_nH_{2n}$  or Olefine series, whilst the coal-gases also contain hydrocarbons in which the proportion of carbon to hydrogen atoms is greater than 1 : 2.

My analyses also show that the London gases are still practically free from carbonic anhydride, but that in some of the provincial gases, the amount present is very considerable; thus in the sample from Birmingham it was 1.50, in that from Liverpool 1.70, and in that from St. Andrew's as much as 2.73 per cent. The proportion of nitrogen is also very considerable in some gases, thus in the Birmingham and Ipswich samples it amounted to upwards of 10 per cent.

I have repeatedly heard it stated, even quite recently, that carbonic oxide is usually present to the extent of about 10 per cent in coal-gas. Now that is certainly not the case, for I have never yet examined a sample of gas containing that amount. It would be much nearer the truth to say that the proportion of carbonic oxide usually varies from 3.5 per cent. My analyses of coal-gas were, however, especially undertaken with the view of tracing the relationship between chemical composition and illuminating power. As it was quite impossible to take the illuminating power of these numerous gases at the time of collecting the various samples, I have been compelled to adopt the reputed illuminating power given in the Gasworks Statistics edited by Mr. C. W. Hastings.

Now, the points in the chemical composition of the gas which are of primary importance in determining its illuminating power are: firstly, the proportion of hydrocarbons,  $C_nH_m$ , which the gas contains; secondly, the equivalence in ethylene of these hydrocarbons; and thirdly, their carbon and hydrogen density respectively. The other ingredients of the gas, generally amounting to upwards of 90%, are of only secondary importance in influencing the illuminating power.

Taking in the first place the proportion of hydrocarbons  $C_nH_m$ , it will be seen that, roughly speaking, the illuminating power rises or falls as the proportion of these hydrocarbons increases or diminishes. Many instances will, however, be seen of a gas with a higher illuminating power possessing a lower percentage of these hydrocarbons than another gas with an inferior luminosity. Far more faithfully does the proportion of ethylene to which the hydrocarbons are equivalent accord with the illuminating power. In fact, it will be found that every 1 per cent. of ethylene is roughly equivalent to from 2.3 candles illuminating power. This is indicated in the column entitled "Ratio of illuminating power to equivalent percentage of ethylene." The ratio in the case of cannel-gas is about 2, whilst for the coal-gases it is somewhat greater, and in a few instances amounts to 3. This difference in ratio depends mainly upon the difference in carbon density of the hydrocarbons, i.e., where the ratio is great the carbon density is generally great also and *vice versa*. This shews that the hydrocarbons of high carbon density possess a greater illuminating power than is indicated by their equivalent in ethylene. Thus 1 volume of benzene vapour is, as regards carbon vapour, equivalent to 3 volumes of ethylene; but the illuminating power of benzene, I shall presently shew you, is not three times but nearly six times as great as that of ethylene, for by adding ethylene to coal-gas that had been disilluminated by means of bromine, I found that it required no less than 13.41 per cent. of ethylene to obtain a gas of 16.84 candles illuminating power, whilst 3.09 per cent. of benzene vapour already imparted to the disilluminated gas an illuminating power of no less than 22.92 candles. Thus

1 per cent. ethylene = 1.26 candles.

1 " benzene vapour = 7.42 candles.

So that the benzene vapour has an illuminating power almost six times as great as that of the ethylene. Indeed it is this disproportionate increase in luminosity of the hydrocarbons as their carbon density rises, together with our inability to determine the individual hydrocarbons composing the group  $C_nH_m$ , which prevents our being able to calculate with absolute precision the illuminating power of gas from analyses such as those to which I have referred.

Since these analytical methods of examination thus fail to afford a complete explanation of the illuminating power of coal-gas, it is obviously of great interest to approach the question by *synthetical* methods. The synthetical enquiry into the source of illuminating power in coal-gas has, until recently, been almost entirely neglected.

This synthetical examination of the subject consists in taking the hydrocarbons of this  $C_nH_m$  group *singly*, and ascertaining by experiment the actual illuminating power which is obtainable from mixtures of these individual hydrocarbons with the several diluents, combustible and incombustible, present in coal-gas.

From the analyses which I have placed before you, it is apparent that one of the main constituents of these hydrocarbons,  $C_nH_m$ , is ethylene,  $C_2H_4$ , which is mixed with smaller quantities of other hydrocarbons, not only of its only class,  $C_nH_{2n}$ , but also of more condensed classes, since in all cases where the hydrogen density has been also determined the ratio of C : H is greater than 1 : 2.

This synthetical examination has only as yet been fully carried out in the case of ethylene, and fragmentarily in that of benzene.

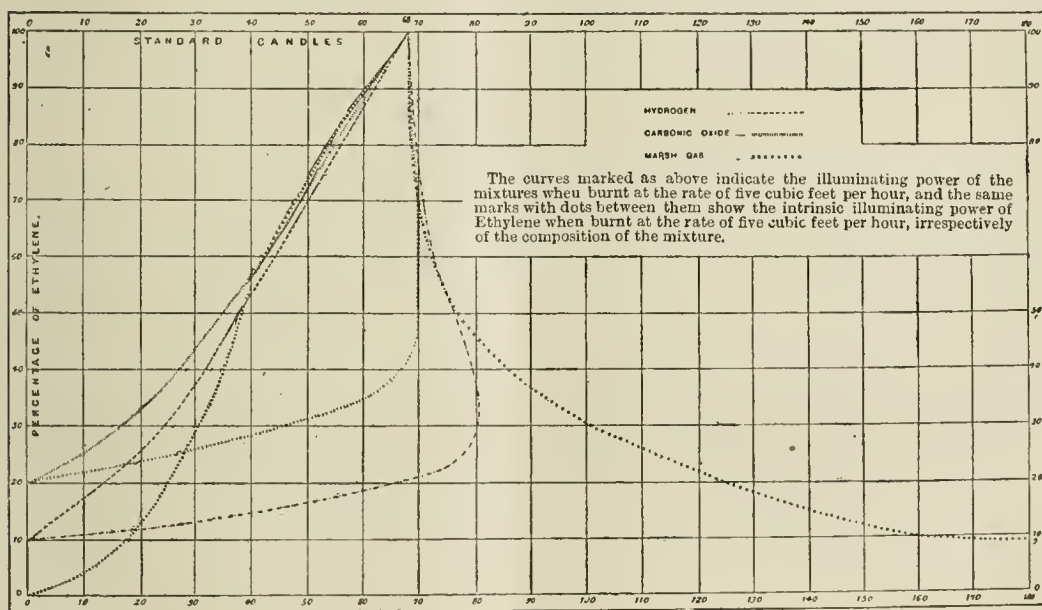
In my experiments with ethylene, I found that the pure gas yields a light of 68.5 candles when burnt from a Referee's burner, and calculated for a consumption of 5 cubic feet per hour. By admixture

with any diluent, excepting oxygen, this illuminating power of the ethylene is gradually reduced. Let us consider the different ways in which these several diluents operate in reducing this illuminating power.

In the first place, taking the combustible diluents—hydrogen, carbonic oxide, and marsh-gas. These three diluents reduce the illuminating power of the ethylene by practically the same amount, as long as none of them form more than 40 to 45 per cent. of the mixture. When, however, the proportion of diluent exceeds this amount, the difference between them becomes more manifest; of the three, carbonic oxide is found to reduce the illuminating power most, and marsh-gas least. Thus, mixtures of ethylene and carbonic oxide cease to have any practical luminous effect when the proportion of the former does not exceed 20 per cent. of the whole. Mixtures of ethylene and hydrogen, on the other hand, cease to be luminous when the proportion of ethylene is less than 10 per cent. of the whole. Whilst all mixtures

of ethylene with marsh-gas possess some illuminating power, for even marsh-gas alone yields a very perceptible amount of light when consumed from a Referee's burner. In order to illustrate this difference in the effect of the several diluents in coal-gas, let us take an example. The gas supplied by the Gas Light and Coke Co., as we see from the table, contains the equivalent of 6.58 per cent. of ethylene. If this proportion of ethylene were mixed with hydrogen only, we see from the diagram of curves that it would yield practically no light at all, if mixed entirely with carbonic oxide it would be similarly non-luminous, whilst if mixed with marsh-gas alone it would yield a light of some 13 or 14 candles. As a matter of fact, we see from the analysis that it is actually mixed with only 37.64 per cent. of marsh-gas, whilst with the other constituents of the gas it can yield no light at all; so that in the actual gaseous mixtures, indicated by the analysis, the 6.58 per cent. of ethylene could not yield as much light as 13 to 14 candles; and in fact, I find that with disilluminated coal-

Illuminating power of Ethylene when mixed with Combustible Diluents.



gas (Gas Light and Coke Co.) it requires about 13 per cent. of ethylene to restore the 16-candle illuminating power, whilst if marsh-gas were the only diluent in coal-gas, it would require less than 10 per cent. of ethylene.

It is very evident, therefore, that the nature of the combustible diluents is by no means a matter of unimportance, as has often been supposed. If to secure the greatest illuminating power is the object of the gas manufacturer, then the larger the proportion of marsh-gas, the greater will be the light obtained for the smallest amount of hydrocarbons.

That marsh-gas is not only the best diluent for ethylene, but also for other hydrocarbons, is seen from the experiments of Frankland and Thorne, who found that marsh-gas saturated with the vapour of benzene yielded decidedly more light than either hydrogen or carbonic oxide saturated in the same manner.

A remarkable feature connected with the dilution of

ethylene by non-luminous combustible gases is that the intrinsic luminosity of the ethylene remains practically constant until the proportion of diluent amounts to about 50 per cent. of the mixture; that is to say, if the illuminating power of the mixture be calculated for a uniform consumption of 5 cubic feet of ethylene per hour instead of 5 cubic feet of the mixture, the illuminating power of pure ethylene, viz., 68.5 candles, is maintained. This intrinsic luminosity of ethylene under dilution is indicated in the diagrams by the dotted curves. From these curves it will be seen that the further dilution (beyond 50%) of the ethylene has a very different effect upon its *intrinsic* luminosity, according to the diluent employed; thus, in the case of carbonic oxide there is a rapid decline in the case of hydrogen, a slight increase quickly followed by speedy decline, whilst in the case of marsh-gas a continuous and rapidly increasing augmentation in the intrinsic luminosity of the ethylene.



If we now turn our attention to the incombustible diluents in gas, we shall find that in many respects their influence upon the illuminating power is very different from that of the combustible diluents. The incombustible diluents of coal-gas are carbonic anhydride, aqueous vapour, nitrogen, oxygen, and atmospheric air. My experiments with these gases have been chiefly confined to a study of their influence upon the illuminating power of ethylene.

Carbonic anhydride, aqueous vapour and nitrogen, all produce effects which are alike in kind, but different in degree. All three exert a prejudicial effect upon the illuminating power, but this effect is greatest in the case of carbonic anhydride—least in that of nitrogen. My experiments with aqueous vapour were naturally limited to determining the difference in illuminating power of dry gas and gas saturated with moisture at the ordinary temperature of the air ( $20^{\circ}\text{C}$ ). The mean of several experiments, both with coal-gas and ethylene, showed that the presence of 2 per cent. of aqueous vapour reduces the illuminating power of the gas by about 3 per cent.

Reference to the diagram of curves will show that carbonic anhydride effects complete disillumination

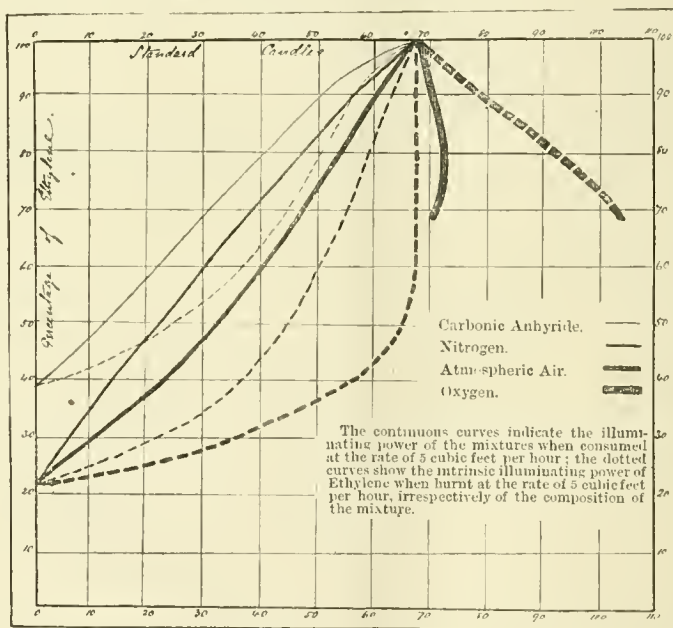
of the ethylene when it is present to the amount of 60 per cent.; similar disillumination is not effected by nitrogen until its amount reaches between 70 and 80 per cent.

The curves indicating the intrinsic luminosity of the ethylene will be seen to be entirely different from those which were obtained in the case of the combustible diluents; for instead of the intrinsic luminosity remaining unaffected by dilution of the ethylene up to 50 per cent., it is, in the case of carbonic anhydride and nitrogen, rapidly reduced from the beginning.

Oxygen, on the other hand, produces on the illuminating power, a very different effect from any of the other diluents, either combustible or incombustible, for it not only greatly increases the intrinsic luminosity of the ethylene, but the mixtures of ethylene and oxygen possess a greater illuminating power than the pure ethylene itself. The experiments with oxygen can unfortunately not be pushed very far, on account of the danger of explosion.

As might be anticipated, the action of air is intermediate between that of oxygen and nitrogen. Thus the illuminating power is reduced by admixture of

Illuminating power of Ethylene when burnt with Incombustible Diluents.



air, but the intrinsic luminosity undergoes no diminution until the proportion of air reaches about 50 per cent. Indeed, it very much resembles, in its effect, the combustible diluent carbonic oxide. Complete disillumination is effected by between 70 and 80 per cent. of air as of nitrogen.

It is not difficult to give an explanation of these phenomena. In the case of carbonic anhydride, aqueous vapour and nitrogen, the illuminating power is reduced, owing to the temperature of the flame being reduced. In the case of nitrogen, this reduction in temperature is due to one cause only, viz., the heat required to raise the inert nitrogen to the temperature of the flame; in the case of carbonic anhydride and aqueous vapour, there is the same cause of diminution, and also another, viz., the reduction of carbonic anhydride to carbonic oxide on the one hand, and the dissociation of aqueous vapour on the other, both of which changes are accompanied by the absorption of a large amount of heat, and

consequent diminished flame-temperature. Thus the greater refrigeration in the case of carbonic anhydride, than in that of nitrogen, is accompanied by a greater destruction of illuminating power.

In the case of oxygen, the greater illuminating power is occasioned by the higher temperature of the flame; there is, however, another agency operating in the contrary direction, and this is the more rapid oxidation of the illuminating material, which takes place when the gas is mixed with a supporter of combustion. If mixtures containing larger proportions of oxygen could have been examined with safety, it would doubtless have been found that this increased luminosity, or "super-illumination," as I have designated it, would have been followed by a reduction and eventual extinction of the illuminating power.

When a suitable proportion of air is added to the gas, the temperature of the flame is increased, but not nearly to the same extent as in the

case of oxygen; there are, however, two other agencies which also come into play, and tend to diminish the illuminating power; these are—firstly, dilution of the gas with the inert nitrogen (this factor is wholly absent in the case of the oxygen above); and secondly, more rapid oxidation of the illuminating material. The two latter factors produce, however, an effect greater than the first, so that, on the whole, the illuminating power is reduced, although until the air is present to the extent of about 50 per cent., the intrinsic luminosity remains unaffected, but as the proportion of air increases beyond that, its prejudicial effect approaches that of nitrogen, which it equals at the point of complete disillumination. It has, moreover, been experimentally shown that the reduction in the temperature of the flame by dilution with atmospheric air, gradually approaches that which is caused by dilution with nitrogen.

Of what practical value to the gas manufacturer are the experimental facts and theoretical considerations which have been discussed above?

In the first place, we see that neither is the proportion of heavy hydrocarbons in coal-gas, nor the equivalent in ethylene of these hydrocarbons, a definite measure of the illuminating power of the gas. For the same amount of light may be yielded by a small proportion of a heavy hydrocarbon, like benzene, as is yielded by a proportion of a lighter hydrocarbon, equivalent to more ethylene than the benzene. Thus the London gas of the Gas Light and Coke Company, with its heavy hydrocarbons, equivalent to between six and seven per cent. of ethylene, gives as much light as the same gas with these hydrocarbons exchanged for 13 per cent. of ethylene. Thus by increasing the density of the hydrocarbons, the same light can be obtained by means of a much smaller proportion of hydrocarbons, so that if the heavy hydrocarbons present in any given gas (which, as the analyses show, are nearly wholly olefines), could be converted into hydrocarbons of the acetylene and benzene series, the illuminating power of that gas would be greatly increased, without importing into it any *foreign* illuminating constituents. Unfortunately we are at present still very much in the dark as to the precise conditions of temperature, etc., influencing the conversion of hydrocarbons from one series to another.

In the second place, as to the combustible diluents with which the hydrocarbons are mixed, it is obvious from my experiments that marsh-gas is the one most fitted for developing the illuminating power; and, indeed, when an Argand burner is used, it cannot be regarded as a diluent only, but must be classed with the illuminating constituents of the gas. It must, however, be remembered that marsh-gas also possesses disadvantages as a diluent, for it consumes four times as much oxygen as does the same volume of hydrogen, and produces three times as much heat, besides producing its own volume of  $\text{CO}_2$ , so that it tends to vitiate the atmosphere of the room in which the gas is burnt. From my experiments, it also appears that hydrogen is a more advantageous diluent than carbonic oxide.

Lastly, as regards the incombustible diluents which are present in gas, it appears that these should be conspicuous by their absence; for although oxygen itself increases the illuminating power, yet in gas it is always present, with an excess of nitrogen over and above that required for the proportion in atmospheric air, so that its presence can only be accompanied by loss of light. Of the incombustible diluents, carbonic anhydride is the one which is most, and atmospheric air least prejudicial to the illuminating power, whilst water-vapour and nitrogen

are intermediate in their action. Carbonic anhydride is now fortunately almost banished from the gas supplied to many of our large towns, whilst nitrogen is allowed to exist, sometimes in no small proportion.

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## SOME NEW PHASES IN PHOTOMETRICAL PRACTICE.

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THE attention which has been paid within the last few years to the improved illumination of open spaces and large areas generally, and the introduction of the electric light, and gas burners of high power, has inevitably led to a reconsideration of the methods in use for estimating the value of the various systems adopted.

It was formerly considered sufficient to estimate the intensity of the luminous rays in a horizontal direction only, irrespective of the value of those rays which are actually utilized in practice. Such a system was doubtless useful in those cases in which burners of similar primary construction were employed; but with the various forms of burners and lanterns recently introduced to the public, such a system is entirely erroneous, and can only afford results of a misleading character.

In order to ascertain the true value of a luminous agent, it is necessary to determine the power of those rays falling at angles, varying from the horizontal to the vertical, or more strictly, through the whole of the semi-circle, from the vertical line above to the vertical line below the point of illumination, thus—



For this purpose the ordinary form of photometer is altogether unsuitable, and can only be employed after considerable modification, and with an expenditure of time and labour, which is all but out of the question. I have therefore devised an instrument of entirely different construction to the usual form, which renders the testing of the angular rays both easy and rapid.

Before proceeding to the description of this photometer, it will be advisable to discuss the principles adopted, and the reasons for them.

When the Committee of the Gas Section of the International Gas and Electric Exhibition, held at the Crystal Palace last year, invited Prof. William Foster and myself to report upon the various burners exhibited, one of the first points considered by us, at the request of the committee, was the estimation of the angular rays emitted from the various burners submitted to our examination. For this purpose we employed a small portable photometer designed by Mr. F. W. Hartley, and termed by him the "Universal" photometer. The instrument consists of a light narrow table, 11 inches wide, 2 feet 6 inches high, and 5 feet 6 inches in length. The scale is divided into inches and tenths, and is 21 inches in length. It is fitted into, and capable of being shifted and fixed at any position within a groove in the table top, which has a long slot along its centre, below which slot is a brass socket connected by wire cords passing over pulleys to the winch handle, similar to the arrangement in the Evans photometer for moving the candles, and serving the same purpose, viz., the movement of the standard. The disc carrier is supported on a stand, the base of which is fitted with a pointer or index coinciding with the vertical line of the disc. The



disc carrier, like the scale, may be shifted along the table, and both must be shifted at the same time, the index of the disc carrier and the zero of the scale always being made to coincide with each other. With the photometer a strong sliding pillar is provided,

which, like the photometer, stands upon the floor, and is provided with levelling screws and plumb-lines. This pillar serves to carry gas burners, or lamps of various size, as required. The apparatus, ready for use, is shewn in the following woodcut.

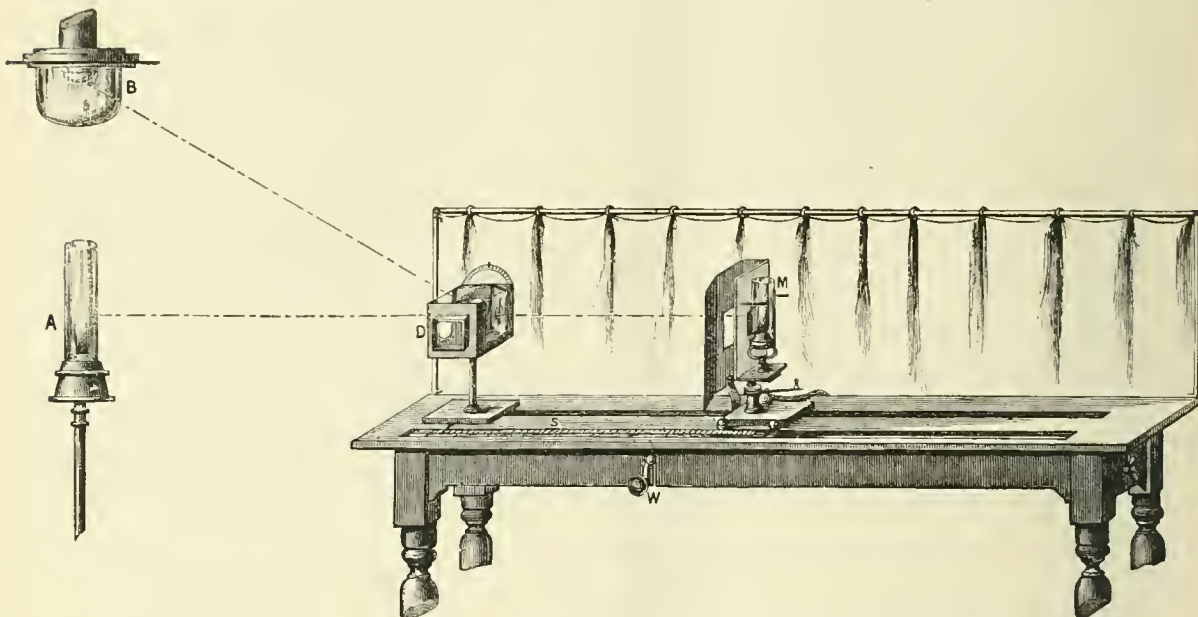


Fig. 1.

Mr. Hartley has calculated a series of tables for use with the instrument, by means of which results are readily obtained.

As submitted to us, the disc was rigidly fixed in the usual vertical position, but at our request this was so arranged as to be susceptible of adjustment at any required angle, so that the rays from the standard and the burner under examination, whatever its position, might impinge upon the screen at equal angles. The following considerations will shew our reasons for this:—

cumference of a circle, whose centre is coincident with the centre of the disc, the number of rays impinging upon the unit area of the disc must be less, and continue to decrease, as the position of the light is increased from that of the horizontal line; and this decrease is in exact ratio to the cosine of the angle formed by the position of the light with regard to the disc and the path of horizontal rays. Therefore the number of rays impinging upon the vertical disc will diminish with the cosine of the angle thus formed. The following diagram shews this very clearly.

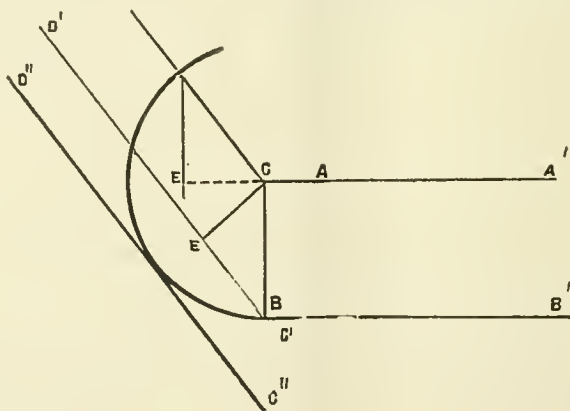


Fig. 2.

When two lights are opposed to each other in a horizontal direction, and a vertical screen is placed between them, it is evident that the rays impinging thereon must do so in accordance with the well-known law of the squares of the distance. If the actual distance of one of the lights from the screen remains constant while travelling through the cir-

Let  $AA^1 BB^1$  represent the section of horizontal rays impinging at right angles upon the vertical disc  $CC^1$ , and  $CDC^1D^1$  the section of an equal number of rays thrown at a downward angle from a source of light placed above the horizontal. It is evident that the whole of the angular rays do not impinge upon the disc  $CC^1$ , but that the rays which do so are defined

by  $CDC^1D^1$ . By drawing the circumference of a circle whose radius is  $CC^1$ , and finding the cosine  $CE$ , it is at once seen that the section of the rays  $CF$ , which impinge upon the disc, is in exact proportion to the cosine of the angle of incidence  $CE$ .

When the light is raised throughout a quadrant, the number of rays impinging upon the vertical disc will be *nil*, and thus, although the burner may be one of high illuminating power, such a system of photometry would fail to record any value for it.

Another important point in connection with the vertical disc must not be overlooked, and that is, that when the rays of light impinge upon a surface at an oblique angle, a considerable loss of light occurs by reason of the increase of reflection and absorption, which preponderates over the loss incurred when the angle of incidence forms a right angle. This loss increases with the increase of the angle, and seriously vitiates any results obtained.

Table I shews the results of some tests made in this manner.

TABLE shewing the Illuminating Power of *Angular Rays* when tested with the Photometer Disc fixed in a vertical position, and when it is arranged so that the angles of incidence are identical:—

#### RAYs FROM BURNER $22.5^\circ$ WITH HORIZONTAL LINE.

Readings with Disc Vertical.	Corrected for Cosine of angle = $.9239$ .	Readings with Disc arranged for equal angles of incidence.	Loss % by estimation with Vertical Disc, due to reflection.
38.7 .....	41.9 .....	44.1 .....	4.9
205.0 .....	222.0 .....	245.0 .....	9.4
453.5 .....	491.0 .....	519.5 .....	5.5
27.0 .....	29.2 .....	29.7 .....	1.6
326.0 .....	353.0 .....	352.0 .....	—
62.8 .....	68.0 .....	68.8 .....	1.1
26.5 .....	28.7 .....	30.8 .....	6.8
140.5 .....	152.1 .....	162.2 .....	6.1
			—
Average .....			4.4

#### RAYs FROM BURNERS $45^\circ$ WITH HORIZONTAL LINE.

COSINE =  $.7071$ .

Readings with Disc Vertical.	Corrected for Cosine of angle = $.9239$ .	Readings with Disc arranged for equal angles of incidence.	Loss % by estimation with Vertical Disc, due to reflection.
20.2 .....	28.6 .....	34.9 .....	18.0
282.5 .....	400.0 .....	491.5 .....	18.6
20.6 .....	29.2 .....	34.2 .....	14.6
47.5 .....	67.2 .....	87.3 .....	22.0
23.7 .....	33.6 .....	38.4 .....	12.5
49.4 .....	70.0 .....	85.8 .....	18.4
42.5 .....	60.2 .....	67.1 .....	10.3
15.6 .....	22.1 .....	25.2 .....	12.3
81.0 .....	114.8 .....	136.5 .....	15.9
124.1 .....	176.0 .....	186.9 .....	15.8
104.2 .....	147.8 .....	161.9 .....	8.7
15.7 .....	22.2 .....	25.1 .....	11.6
88.2 .....	125.0 .....	144.0 .....	13.2
14.5 .....	25.5 .....	25.7 .....	0.8
9.2 .....	13.0 .....	12.7 .....	—
129.3 .....	183.0 .....	207.0 .....	11.6
9.0 .....	12.7 .....	12.6 .....	—
52.2 .....	74.0 .....	86.5 .....	14.5
			—
Average .....			12.15

#### RAYs FROM BURNER $67.5^\circ$ WITH HORIZONTAL LINE.

COSINE =  $.3827$ .

Readings with Disc Vertical.	Corrected for Cosine of angle = $.9239$ .	Readings with Disc arranged for equal angles of incidence.	Loss % by estimation with Vertical Disc, due to reflection.
55.3 .....	144.5 .....	378.0 .....	61.8
82.8 .....	216.5 .....	920.0 .....	76.5
			—
Average .....			69.1

After correction for the diminished number of rays impinging upon the disc at the different angles, the value obtained is deducted from that found by estimation with the disc arranged for equal angles of incidence, and the difference between the two results calculated into percentages. By this means I find that when the burner is at an angle of  $22.5^\circ$  above the horizontal, the average loss due to reflection from the vertical disc is 4.4 per cent. ; at  $45^\circ$  it is 12 per cent. ; and at  $67.5^\circ$  69 per cent.

It is obvious, therefore, that the method of estimating the illuminating power of angular rays by means of a vertical disc is erroneous.

By arranging the disc so that the angle of incidence is equal on either side, thus—

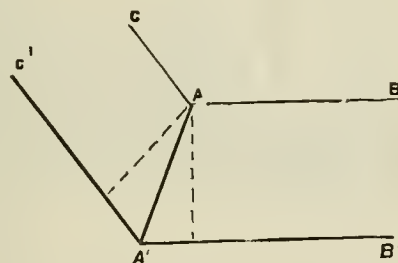


Fig. 3.

Disc  $AA'$ . Horizontal rays  $ABA'B'$ . Angular rays  $CAC'A'$ .

we equalise both the proportionate number of rays impinging thereon, as well as the loss due to reflection. Determinations thus made possess all the value of those made with a vertical disc and horizontal rays on either side in the usual manner.

For the purpose of comparison with the "Radial" photometer, as I have termed my design, I append the following woodcut of a portable photometer, made by Messrs. Sugg & Co., which embraces all the essential points of the most approved pattern of the ordinary bar photometer. It is easily taken to pieces and packed in a box for conveyance and is readily fitted up for use, with a little practice, in five minutes.

The principle involved in the construction of the Radial photometer is very simple, viz. : that the light under examination should be rigidly fixed in one position while the estimations of the value of the angular rays emitted from the horizontal to the vertical, either above or below, are being made, thus ensuring perfect steadiness of the burner, or other luminous point.

The apparatus consists of two vertical supports, one of which is permanently fixed to the base-board or foot, while the one on the right hand travels on rollers on the base-board in such a position that it will run in front of the fixed support.



The two uprights are connected by a bar, the ends of which work upon trunnions, or axles, attached to blocks which travel in the grooves of the uprights. These blocks can be clamped in any desired position. One end of the bar is attached to the front of the fixed upright, while the other end is attached to the travelling upright at the back, so that when the two uprights are in juxta-position the bar is perpendicular between them. The centres

of the trunnions correspond in position with the centres of the two graduated dial plates in front of the uprights. The distance between the centres of these dial plates is 50 inches. It is therefore evident that whatever position the bar may be in, the distance from the centre of one dial to that of the other must be constant. In front of the dial plate on the travelling upright the screen or disc-holder is fixed, so that its centre is coincident with the centre of the dial.

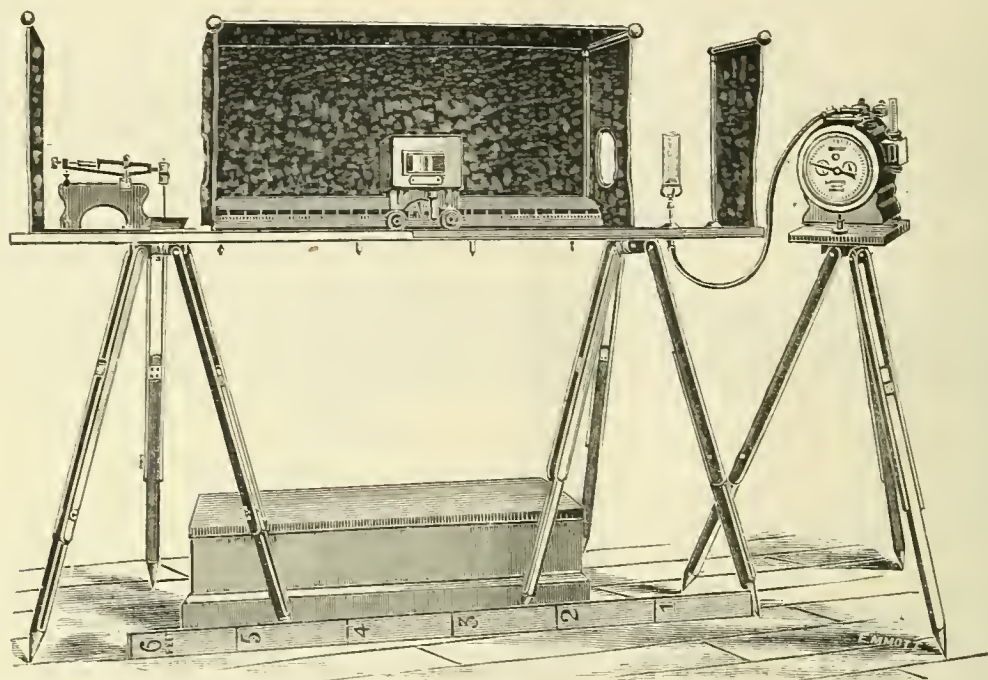


Fig. 1.

Attached to the block in the groove of the travelling upright support is the horizontal bar carrying the standard.

The standard is supported in front of the horizontal bar by a travelling carriage, working on rollers, and is moved by a cord and winch, conveniently placed on the right-hand side of the graduated dial on the support. Attached to the block carrying the photometer disc is a brass rod, which is brought well forward and then curved round for the purpose of carrying a velvet curtain to screen off extraneous light when readings are being taken.

The two dial plates are graduated; the larger one on the fixed support in degrees, and the smaller one on the travelling support in half degrees, which are numbered as whole degrees for the purpose of facilitating the setting of the disc for equal angles of incidence, so that when the bar is set, say, at 40 degrees, the disc-pointer is to be set at 40, it will then be in the proper position, viz., 20 degrees. The disc may be arranged to work automatically with the movement of the bar by means of a simple mechanical appliance, so that whatever may be the position of the bar, the disc will be at the correct angle.

A brass rod is provided for adjusting the position of the burner, &c., to be tested. It has to be pushed through the centre of the block and trunnion on the fixed upright support, and will then be at right angles with the plane of the dial, and project exactly through its centre, by which means it is easy to fix the exact position of the flame in front of the apparatus. The light to be tested may be brought forward to the full

extent which can be attained by the disc and standard, which, obviously, can be regulated as desired, so that the size of the burner or lantern—which may be tested with this apparatus—is practically unlimited, due regard being paid to the length of the bar and the power of the light.

When a test is commenced the light to be examined is fixed on the support attached to the block in the fixed upright, and accurately centred with the dial plate, which is to be lowered to the bottom of the groove in the support. The block in the travelling support has next to be raised, which operation will bring it immediately over the burner, the travelling upright being in front of the fixed support, and the pointer on the bar indicating 90° on the large dial plate. The photometer disc is to be arranged for equal angles of incidence, by turning it until its pointer is at 90, when a reading can be taken. The clamp holding the top block in position is then loosened, and the handle working the rack and pinion of the travelling support turned until the bar is at an angle of 80°, the block must then be clamped, the disc adjusted to 80°, and so on for each degree or ten degrees as desired, until the horizontal rays are estimated. The block supporting the light is then to be raised to the higher position, and the bar adjusted for the desired angle below the horizontal, and a second series of readings taken until the downward vertical rays are estimated.

The following wood-cut shews the instrument arranged for testing the rays thrown downward at an angle of 45°

The Bunsen, or greased disc, under ordinary circumstances, with lights of equal colour, is all that can be desired; but when used for testing the electric light or gas burners of the recuperative class, it is very unsatisfactory, and at times useless, in consequence of the great difference in tint between the light emitted from the standard and that of the burner under examination. I have, therefore, abandoned its use for these purposes, and use a modified form of the Leeson or "Star" disc. As originally designed, this disc was unsatisfactory, in consequence of the "cockling" of the two thin papers on either side of the perforated stout paper. To such an extent was this fault found to interfere with the readings, that the Gas Referees, some time back, disallowed its use at the Metropolitan gas testing stations under their charge.

Finding that the Bunsen disc did not answer all the purposes required, I modified the Star disc by pressing together the three papers, of which it is formed, with very thin starch water, and drying the moist disc under pressure. This treatment effectually prevented "cockling," and the use of the disc in its present form is sanctioned by the Gas Referees.

The great advantage of this form of disc is that very sharp readings are readily obtained, with totally different coloured lights; red and blue lights are compared with the greatest ease, an advantage which no words of mine can enhance.

It is to be hoped that in future all comparative tests of the value of various burners will be so conducted as to shew the actual work done by them, not only in one direction, but in all directions. With argand and other circular burners, this can be done

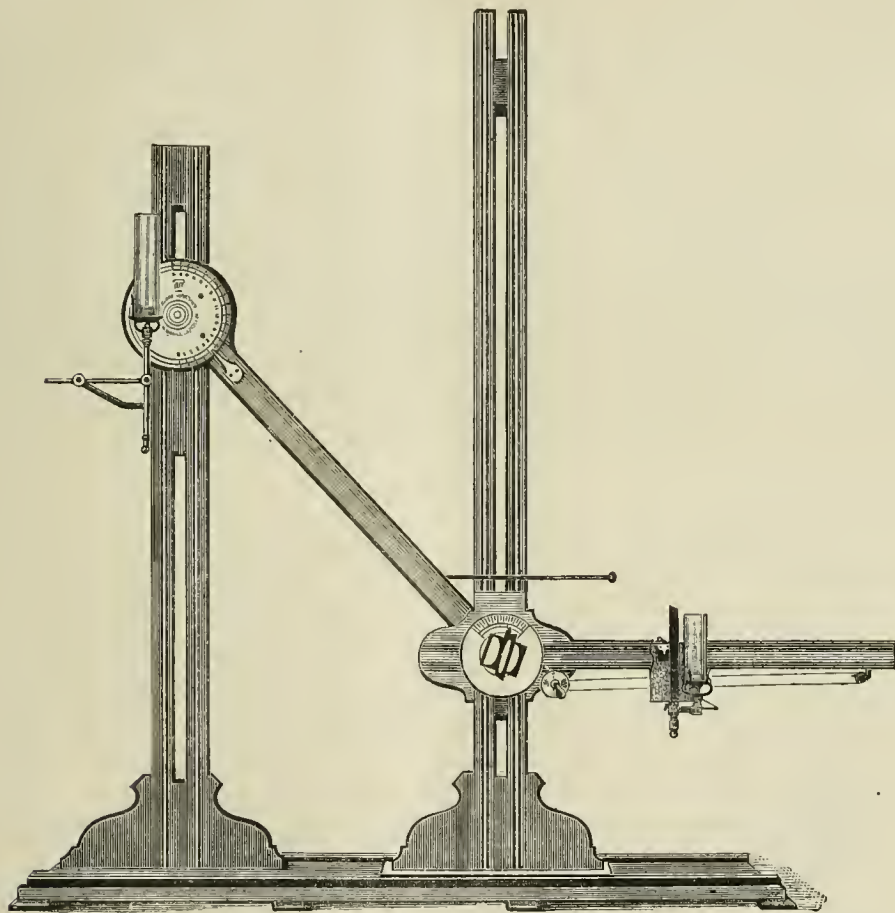


Fig. 5.

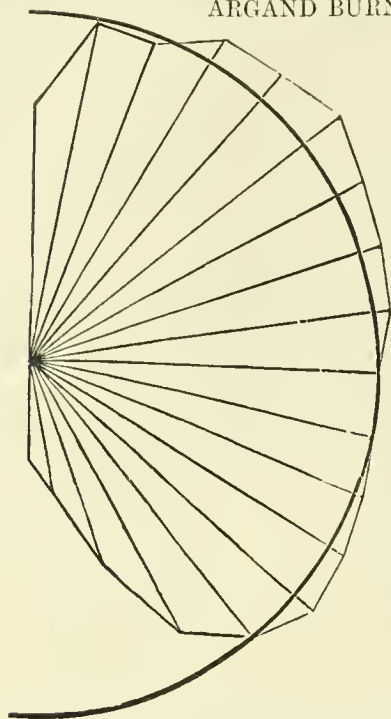
by making one series of tests from the vertical above to the vertical below, at every 10 degrees. But in the case of flat-flame burners, it is necessary that this series should be made in duplicate, one with the flame flat, or at right angles to the bar of the photometer, and one with the flame placed with its edge to the bar. An extensive series of experiments on this point has shewn that very considerable differences exist between the quantity of light emitted from the flat surface and from the edge of various burners; this difference varying from 10 to 35 per cent. of the light emitted from the flat surface. Therefore, it is very necessary that the two series of tests should be made

and an average taken, which should be held to represent the value of the burner.

For the purpose of facilitating comparison, I have drawn the following diagrams, representing the quantity of light emitted in the different directions, by which it will be seen that the ordinary method of testing burners does not give really comparative results of any value, as the horizontal rays in each case are curiously less in illuminating power than the angular ones, both above and below them; therefore horizontal testing only is unfair to the burner, and in all competitive trials should be supplemented in the manner I have described.



## ARGAND BURNER.



The thick line represents the circumference of a circle whose radius equals the value of the light thrown from the burner horizontally.

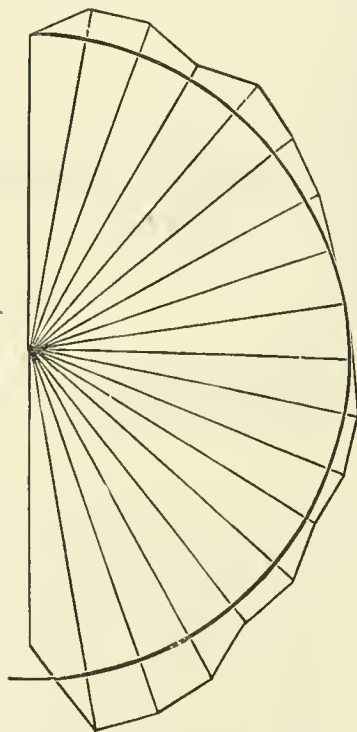
I am aware that I am proposing a complete revolution in practical photometry, but, having shewn a full justification for it, I venture to hope that those experienced will readily agree with me, that the sooner the old erroneous methods are abandoned the better.

By means of the radial photometer, and the improved Leeson's disc any light can be readily tested in a most satisfactory manner, and results obtained which will fully repay the small amount of additional work expended in their production.

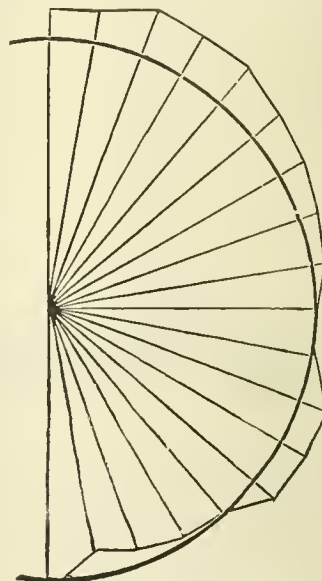
The importance of a reliable standard of light is admitted by all experts in photometry, and a great deal of work has been done by various investigators with the view of obtaining something of a more definite character than candles. It is to be hoped that before long a change from the present parliamentary standard will be authorised, and one or more of the proposed substitutes for candles made legal.

Enough has been done to prove that the Pentane test of Professor A. Vernon Harcourt, and the screen of Mr. Methuen possess all the requirements of practical standards; and where expense is no object in the one case, or gaseous fuel is obtainable in the other, and a small degree of illumination is desirable, little objection can be made to them. But where gaseous fuel is not at hand, and when a standard of light of higher illuminating power is desired, no better standard can be advised than the sperm oil lamp of the late Mr. T. W. Keates, which was fully described by him in the *Journal of Gas Lighting* of March 16, 1869. At that time the lamp was arranged to give a light equal to 10 candles, but since then it has been improved so as to yield a light equal to 16 candles, which can be modified, when desired, to give a light of two, five, or ten candles, by simply cutting off

## FLAT FLAME BURNER No. 1.

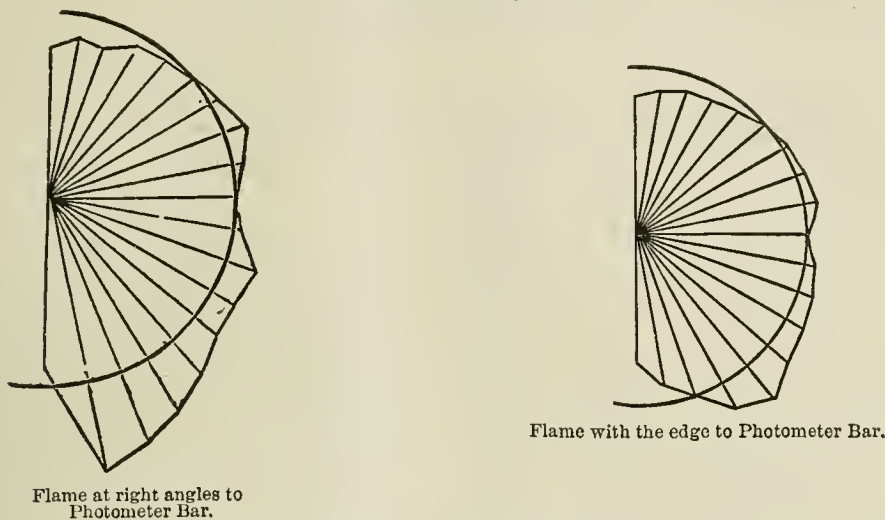


Flame at right angles to Photometer Bar.



Flame to the edge with Photometer Bar.

FLAT FLAME BURNER, No. 2.



portions of the flame by means of a suitable screen ; in which case sufficiently accurate results for ordinary purposes can be obtained without weighing the oil.

In raising this question of the lamp, I am perfectly aware that many will think that I am “thrashing a dead horse.” In answer to those who do so, I will ask for any systematic tests made with this lamp which shall prove its unreliability. The objection that the lamp is used with a wick I hold to be utterly unscientific and unworthy of consideration in the face of the fact that, when properly used, the lamp yields a light which is practically constant. In proof of this statement, I would point to the following series of tests of a gas flame arranged to yield a steady column of light, 3 inches in height, by cutting off the light from the top of the flame with a screen, so as to exclude errors, as far as possible, arising from slight variation of consumption or quality of the gas.

Table II. shews the results of some preliminary tests made in this manner.

TABLE II.

*Shewing the results of tests of the angular rays emitted from an argand, and from two flat flame burners of different construction. The photometer disc was arranged so that the angle of incidence was equal on either side. Results stated in percentages of maximum intensity.*

Rays from Burner.	Argand.	Flat Flame, No. 1.		Flat Flame, No. 2.	
		Flame at right angles to Photometer Bar.	Flame with the edge to Photometer Bar.	Flame at right angles to Photometer Bar.	Flame with the edge to Photometer Bar.
90° above horizontal .....	64·2	84·8	79·6	56·0	50·0
80°   "   "   " .....	94·3	94·2	79·6	60·0	52·0
70°   "   "   " .....	86·4	95·6	84·0	60·0	55·0
60°   "   "   " .....	96·5	92·7	84·0	64·0	56·0
50°   "   "   " .....	97·1	95·6	83·2	66·0	58·0
40°   "   "   " .....	100·0	91·3	79·6	68·0	60·0
30°   "   "   " .....	97·1	87·0	76·8	72·0	62·0
20°   "   "   " .....	96·5	84·0	75·3	74·0	63·0
10°   "   "   " .....	95·0	84·0	74·0	70·0	63·0
Horizontal .....	90·0	84·8	71·0	68·0	60·0
10° below horizontal .....	90·0	89·1	71·6	72·0	65·0
20°   "   "   " .....	92·9	89·1	76·8	78·0	68·0
30°   "   "   " .....	94·3	87·6	76·8	76·0	69·0
40°   "   "   " .....	97·1	94·2	76·0	78·0	72·0
50°   "   "   " .....	91·5	93·5	69·5	84·0	76·0
60°   "   "   " .....	81·5	98·5	68·1	88·0	70·0
70°   "   "   " .....	57·2	100·0	66·7	94·0	60·0
80°   "   "   " .....	36·5	99·2	63·8	100·0	56·0
90°   "   "   " .....	26·4	74·0	72·1	62·0	45·0



TABLE III.

*Shewing the results of tests made with Keates' lamp, of a flame 3 inches in height, the light from portions above that height being cut off by means of a screen.*

	Light given by Standard Lamp, corrected for consumption of Sperm.	Ascertained value of Gas Flame.
1st day	16.55 .....	18.03
"	16.82 .....	17.85
"	16.89 .....	18.07
2nd day	15.50 .....	17.99
"	16.21 .....	17.94
"	16.75 .....	17.82
"	16.38 .....	18.32
"	17.02 .....	18.21
3rd day	16.48 .....	18.06
"	16.68 .....	18.11
4th day	16.10 .....	17.93
"	16.50 .....	18.13
"	17.08 .....	17.93
5th day	16.46 .....	17.88
"	17.00 .....	17.92
"	16.52 .....	18.05
"	17.00 .....	18.06

71% of the tests were *within* 0.1 candle of the mean.

The tests were made at intervals, and on various days, during which, practically, no variation was found in the illuminating power of the gas used. The results may, therefore, be taken as fairly representative of the work done by the lamp.

The conditions under which the lamp must be burnt, are very simple, viz., clean oil and a clean wick. For particulars regarding the oil I cannot do better than refer to Mr. Keates' paper above mentioned. What he said there holds true now, and will hold true so long as there are sperm whales to be caught, and a temperature of 40° F. obtainable.

The only objection of any moment which can be made to the use of the lamp, is that it requires a certain interval of time to elapse between the first lighting and its readiness for use, but as this interval is only about thirty minutes, I do not see that this objection should have weight with those who are desirous of obtaining correct results from an operation which requires such an exercise of patience as photometry admittedly does. It is proverbial that impatient operators make bad photometrists.

The great advantages to be derived from a 16-candle standard, which is ready for use at all times and in all places, are obvious.

In the first instance, the advantage of using a standard which approximates more nearly to the power of the light under examination, must be perceptible to the most superficial observer, as by this means the errors due to the multiplication of the readings by 8, 16, etc., are eliminated, and more correct results obtained.

In the second place, the colour of the standard lamp is almost identical with that of the ordinary 16-candle gas flame, and, therefore, readings with the disc rendered easier.

In the third place, the use of the 16-candle lamp enables estimations to be made of the value of high power gas burners and electric lights, with far greater facility and exactness than can possibly be the case with standards of lower power; with the further advantage of shortening the photometer bar.

I have thus shewn enough to point out the chief advantages of the lamp. The objection that "the lamp will not burn," is so utterly at variance with all the facts, and the results of hundreds of tests made with it, that I pass it over without further comment.

In conclusion, I may state that I do not claim to have settled the many vexed questions arising in photometric practice. On the contrary, the radial photometer was designed to assist in their investigation, and it is to be hoped that its systematic use will throw considerable light on many points connected with the construction of gas burners and other illuminating agents, which at present rest in obscurity. If my endeavours to provide a workable instrument, which shall assist in their solution, meet with any degree of success, I shall be more than satisfied.

#### DISCUSSION.

The CHAIRMAN said the meeting had to thank Dr. Percy Frankland and Mr. Dibdin for their most interesting papers on very important subjects. It was very encouraging to see so many representatives of gas manufacturing industry present; because, the operations concerned acted and reacted upon other branches of chemical industry. Reference to the pages of the *Journal* would show how that fact was recognised, and how frequently the subject was touched upon. One could not help feeling that chemistry had not yet done all it could for the gas industry; and he believed that in the future they would owe a great deal more to its products, besides getting the advantages of the light, for the gas industry had not yet arrived at the perfection which it would some day reach. Then it was wonderful to see how the art of photometry had kept pace with the necessities of the changing lights of the present time; and the points to which their attention had been called were useful, not merely from a photometrical point of view, but also from a practical one. They could not help thinking that it was exceedingly probable a good deal of the complaints of the efficiency and economy of gas-lighting might be owing, in no small degree, to the detestable quality of the burners used, while at the same time it was no doubt possible to use a good burner in such a way as to get a bad result from it. He would, with these preliminary observations, invite remarks from the meeting upon the papers which had been read.

Mr. F. W. HARTLEY said that being an old photometrist he wished to make a few remarks. He believed that he was among the first of those who showed the importance of testing the powers of gas flames at more than one angle; and in the articles which he furnished to the *Journal of Gas Lighting* in January, 1881, he described the photometric arrangement which he used. That arrangement was developed in the "Universal," which was before them, which instrument was employed by Mr. Dibdin and Professor Foster as stated, after the arrangement for the disc to be set at any desired angle had been made. That modification and improvement was entirely due to the gentlemen named; and he hoped that the system of placing the disc at a mid-angle between the unit light and any other which was opposed to it, would be universally adopted, as thereby the true relations in powers were found. He so used the photometer in its later improved state. In his earlier experiments he had, however, sought to ascertain the amount of light which would be received on a perpendicular plane, such as a wall, for instance, where the disc was fixed vertically, as it should be for such purpose, inasmuch as it was clear that the walls of a room could not be turned or twisted to accommodate a light in the ceiling. The angle-screen method was the scientific one; and Messrs. Dibdin and Foster were entitled to the credit of devising it. He was somewhat surprised to hear that when the rays of light fell at an acute angle on the disc, so large a number were, as it were, rendered insensible to the eyes of the observer, seeing that the intensity of

illumination was judged of from the images of the disc in mirrors; but he had no doubt but that Mr. Dibden's statement was quite correct. As regards flat-flames it was the fact that much less light was delivered from their edges than from their surfaces. He had determined the difference with very many gas and paraffin oil flames. If, however, a curved reflector were placed behind a flat flame the horizontal light would be found to be greatest when the edge of the flame was presented to the photometer disc; because, for one thing, the wide flame covered more of the reflector, and prevented it from so fully acting. He regretted that he had not yet worked with Mr. Keates' lamps. That gentleman's lamented death prevented him obtaining it when he had opportunity to try it, and since, he had been too much occupied to find time. He hoped, however, to be able to fully try it presently; but he must differ from the opinion of Mr. Dibdin that a powerful standard of light was necessary, his (Mr. Hartley's) personal experience being that with a unit not exceeding two-candles light the eyes were not so distressed and wearied as when the unit light was powerful; a matter of importance when operations extend over seven or eight hours. With a two-candle unit he had tested lights up to a 1000 candles' power. During the past four months Mr. Heisch and himself had been engaged in investigations on photometric light units, but lamps with wicks were, by instructions, excluded from the inquiry. Mr. Dibdin had shewn them a novel and very beautiful instrument, and he hoped that its employment would lead to the development of many interesting and valuable facts in photometry.

Dr. E. FRANKLAND had been much interested in the paper which had been brought before the Society by Mr. Dibdin. He was not aware before how great the difference could be in the light emitted by argand and fish-tail burners in different directions. It was quite obvious that photometric readings of this kind were absolutely essential if anything like a true result was to be arrived at, and he thought the apparatus which Mr. Dibdin had contrived could be easily worked, and it was obviously susceptible of the attainment of accurate results. He had not quite understood the remarks of Mr. Hartley with regard to the reflection from the paper, but it might be well to bear in mind that the reflection of light from a surface of paper was very different indeed from the reflection of a metallic surface; the reflection from paper increased continually in amount as the angle of incidence formed by the rays and the paper diminished, so that at last, when the rays made a very small angle, a very large proportion of them indeed would be reflected; but how far that would affect the appearance of the disc he was not prepared at the moment to say. Such a photometer would be of essential service in determining the ratio of the illuminating power between the arc lights, which had never yet been properly tested, and he hoped by the help of this instrument they would obtain more trustworthy results than hitherto. With regard to his son's paper, and the experiments he had brought before the notice of the Society, he had not many remarks to offer. It was obvious, as had been pointed out, that there was a very marked difference in the proportion of what might be termed excess-nitrogen in the gases of the different periods mentioned, which might require some explanation. In his own experiments of 1851, it would be seen that the nitrogen was but very slightly in excess of that required by the quantities of oxygen which were also present, whilst the subsequent experiments shewed that the proportion of nitrogen to oxygen had been constantly increasing. He spoke subject to correction by the practical men who were present, but it occurred to him that the fol-

lowing might be the explanation: In 1851, gas was made almost exclusively in iron retorts, and those retorts were much more gas-tight or air-tight than the earthenware retorts since employed. At that time, no doubt the exhaustion was carried on much as it was now—he did not know whether the exhaustion was now greater than it was then—but, at that time, the super-exhaustion, though it would not have the effect of drawing air into the retorts themselves, would draw it in through leaky joints and insufficient seals. There would thus be drawn in the proportions of oxygen and nitrogen contained in the atmosphere. But all the air drawn into a leaky retort would be almost instantaneously deprived of its oxygen, so that if there were a leaky retort into which the furnace gases were drawn, nitrogen, without the corresponding proportion of oxygen, would become mixed with the gas. He hoped the results his son had obtained, in ascertaining the hydrogen density of the illuminating hydrocarbons, would eventually lead to a really trustworthy method of determining chemically the illuminating power of gas. As soon as the intrinsic illuminating power of the different illuminating hydrocarbons, and especially the acetylene, had been determined, he thought a formula might be devised, which would enable the illuminating power of a gaseous mixture to be calculated from the carbon of hydrogen densities with greater accuracy than could be attained by photometric methods. Although it was a method which could never, perhaps, be brought into daily practice, still it would be found very useful to submit gases from time to time to that crucial test. It was not impossible that an apparatus could be contrived, so that by, say, a weekly analysis of the gas of a town, the average quality of that gas could be determined. If a small holder were made to receive a current of the gas, proportional to the amount being supplied at any moment, and then the contents submitted to a weekly analysis, probably a fairly good average would be arrived at, provided they could devise a working formula for the determination of the true illuminating power from the chemical composition.

Mr. DAVID SUGG said:—With regard to placing the discs used in photometry in relative positions to receive the light from the standard and that from the light to be tested equally on both sides, he would quote an instance when his father was engaged making experiments on the Thames Embankment. For this purpose a Richie disc (in the form of an equilateral triangle) was employed. The side towards the standard was fixed, the other made movable, so that it could be elevated into position, making the angles of incidence equal. The images of the discs were received upon a mirror placed above, and at an angle of 45°. It is curious to note, as compared with the results obtained by Mr. Dibdin with his radial photometer, that the observations made by Mr. Sugg with the disc firstly in its normal position and then elevated—making the angles of incidence equal—the readings remained unaltered. This was due, no doubt, to the fact that the same beam of light was received upon the disc, the only difference being the respective angles at which the lights infringed upon it. When short-bar photometers are employed it may be found necessary to have the means of adjusting the disc.

Mr. W. LANT-CARPENTER said it had been pointed out very clearly that some of the remarkable results obtained with regard to the illuminating power of gas, as compared with its chemical composition, were to be explained by the temperature of the flame resulting from the burning of the gas; and he would like to ask whether that line of inquiry had been at all followed out in the direction of investigating the



temperature resulting from the combustion of gas of known compositions. This was an important point, when the constantly increasing use of gas for heating purposes and in gas engines was considered, and it also bore upon the proposal of the late Sir W. Siemens to partially separate the gaseous products of coal distillation into heating gas (the first and last portions) and lighting gas.

Mr. OTTO HEHNER, as one who had to spend a considerable part of his time daily in testing and examining gas, was very glad to hear Mr. Dibdin say that there was every prospect of the present standard of measuring illuminating power being speedily abandoned. There had been much talk of change for a long time past, but we now apparently might hope that not many months would elapse before it was realised. Candles, in fact, were not standards at all—every gas examiner could not but feel that. Could one feel any confidence in the accuracy of the results of one's testing if three successive tests of the same gas, on the same evening, but with different candles, could indicate 15, 16, and 17 candle-powers respectively, when, doubtless, the gas itself had remained practically the same. He did not mean to say that such widely discrepant results were the rule, but still they did occur, and made one feel very diffident in certifying to any slight deficiency in illuminating power of any given gas supply, when one considered that the certificate would probably form the basis of a prosecution against the Gas Company. This absence of a standard worthy of the name made one inclined to regret that Dr. Percy Frankland had bestowed so large an amount of labour in comparing the chemical composition of gas with the nominal illuminating power. If ever a really good standard were obtained, it would be quite impossible to re-calculate Dr. Frankland's results upon that new standard.

Dr. ARMSTRONG remarked that the statements of Dr. P. Frankland afforded conclusive evidence on the subject they were discussing. Professor Frankland had on more than one occasion pointed out what a very bad article the public were supplied with by the gas companies, and he was glad to see his son following in his footsteps and again pointing out the very low quality of the gas supplied to the Metropolis in reference to its illuminating constituents. At the same time his observations indicated the direction in which gas manufacturers must proceed in order to give the public a gas which would satisfy requirements, and be worthy of the present condition of chemical knowledge. He had pointed out, amongst other things, what a great effect a small amount of benzene has upon the illuminating power. They heard on all hands that they were going to recover a very large quantity of benzene and other hydrocarbons which were at present lost in coking coal, and there was little doubt that if they did recover all that was anticipated the hydrocarbons would become drugs in the market. The direction indicated in which they must proceed was perfectly clear, for it was obvious that gas manufacturers might develop their industry to render the gas of sufficient illuminating and heating power by the introduction of volatile hydrocarbons.

Professor FOSTER desired to say a few words with reference to the points which Mr. Hartley and Dr. Frankland had referred to. Mr. Dibdin and himself were rather at a disadvantage on the present occasion; but he had in his hands a report, which had been printed since last November, where the first principles relating to this subject were very fully and fairly discussed, and that discussion, when published, would enable those who took an interest in the matter to better understand the reasons for this new depart-

ture of theirs. The method of obtaining an equality of angle had never, he thought, been adopted before, and there were some points in connection with it to which he would allude. With reference to the use of the disc, light was absorbed, transmitted, and reflected. By so arranging the disc that it makes equal angles with both sources of light, the disposition of the light falling on each side is similar, whatever be the laws with regard to it. That was a fundamental point; and whatever might be the co-efficient of reflection, &c., they would be sure of having trustworthy results by arranging the disc symmetrically with regard to both the lights compared. But he need not now discuss the matter further, because there was no very ready way of understanding what the co-efficient of reflection would be. Mr. Hartley had made an observation about the reflection from the disc, but he had failed, apparently, to notice that the incidence of the reflected rays on either side of the disc would be in the same plane, and that any reflected light as supposed would pass in the way shewn [describing by a diagram]. Mr. Hartley had, therefore, fallen rather into an error in that matter. Whatever the law of reflection might be, the disc was really illuminated and viewed under precisely like conditions on either side.

#### REPLY.

Dr. PERCY FRANKLAND, in reply to the remarks made, said Mr. Foster had spoken of the carbonic oxide and nitrogen, and there certainly did appear to have been some leakage from the large proportion of carbonic acid found. On that point, Prof. Pedler, of Calcutta, had told him the previous week that he had examined the Calcutta gas and found it contained, not only 10 per cent. like the Ipswich and Birmingham gas, but 17 per cent.; so that he supposed the Calcutta gas manufacturers appreciated the advantages of the introduction of air even more than it had been suggested that some of the gas manufacturers in England appreciated it. (Laughter.) Mr. Otto Hehner had regretted that he had unfortunately taken so much trouble with these analyses, when there was no reliable standard of illuminating power; but, of course, he was obliged to be content with what he could get, and no doubt if he had had some of Mr. Dibdin's more reliable standards they would have been of more value. With regard to Mr. Carpenter's remarks on the temperature of flames, he had not investigated them himself, but there were a number of data on the subject, and as far as they were known they coincided very closely with the results he had obtained. For instance, in the case of the combustible diluents the amount of heat produced by marsh-gas was very much in excess of either carbonic oxide or hydrogen. The temperature was mostly reduced by admixture with carbonic anhydride; it was next most reduced by nitrogen, and it was least reduced by atmospheric air; but the reduction in temperature by the admixture of nitrogen and atmospheric air became more and more approximate as the proportion of those diluents increased. As the proportions of nitrogen and atmospheric air increased their illuminating power converged, whilst the reduction in temperature effected by the carbonic anhydride became more than by nitrogen and the atmospheric air. There was a complete accordance, therefore, between the flame temperatures and the illuminating power, so that this matter of the temperature appeared to be the most important of all.

Mr. DIBDIN, in reply, said: With regard to Mr. Hartley's remarks on the high power standard that he had used such a standard and had found a certain amount of strain upon the eyesight from the increased power of the standard, but he himself had worked a

great deal with the 16-candle standard, and though it might be that there was a difference between the eyesight of himself and Mr. Hartley, he must say that after a long day's work with the 16-candle lamp he felt less exhausted than after a similar day's work with the candles; the readings were sharper upon the screens, they took less time to observe, and there was consequently much greater relief for the eyes. On account of the time occupied in trying to balance the position of the disc, when a low power standard was used, one's eyes were tired before the reading could be obtained; but with the 16-candle standard the reading could be taken in a moment; it was as sharp as could be, and the eyes were rested until the next test had to be taken. He had had a great deal of experience with the 16-candle lamp, and he was bound to say that he did not agree with the experience of Mr. Hartley. Mr. Hehner spoke as if he had understood him to say they would actually have another standard of light very shortly, but he was sorry to have given him that impression, as it was, of course, not in his power to determine what the standard should be; that was a matter for Parliament. No doubt many people desired it, but there was a great deal of difference of opinion on the subject of standards, and until it was settled which was the best, Parliament would probably not do anything. It had been suggested that two or three should be allowed by the Board of Trade, to be used if found suitable for the purpose, instead of the present arbitrary rule that only candles should be used. Dr. Frankland had remarked on the subject of the reflection of the light impinging upon the screen at a greater or less angle. He must confess he had felt a little puzzled on that point, and it really required a great deal of work to elucidate the question thoroughly. Until he had this photometer, the testing of the angular rays was a matter of very great difficulty and labour, as at every alteration of the angle the distance had to be calculated and measured, the burner re-adjusted, the photometer scale re-arranged, fresh readings taken of the meter, and in fact each test had to be started *de novo*. It was the anxiety and labour attending all that which induced him to turn his thoughts to such a design as this radial photometer, and he had shewn that by it it was possible to obtain readings at any angle; only one reading of the meter was required, and two settings of the burner—one for the rays above and one for the rays below the horizontal. After the first adjustment only one movement of the burner was required during the whole of the tests from the vertical above, and to the vertical below; and, consequently, the readings taken would be perfectly reliable throughout the whole series, as the consumption could not possibly be affected. He must express his sincere thanks to Messrs. W. Sugg and Co., who had made the instrument for him, for their kind assistance, and the care they had taken in producing the design.

Professor FOSTER resuming, said with reference to the results of Dr. Percy Frankland, the figures as to the illuminating value were more important than some other incidental matter; and still he would like to make a few remarks upon the latter. Some years ago it struck him that the fact of the diminution of carbonic oxide, as shewn by the more recent analyses, might be due to the more perfect methods of closing the retorts. He was not prepared to say whether that was the case or not. Dr. Frankland had alluded to the possibility of air being introduced through the medium of imperfections in the retorts. He (Mr. Foster) might say that he thought there was something underlying the system of purification at Birmingham and Ipswich, where the proportions of nitrogen were shown to be so large, which was slightly different from that adopted

in the majority of cases. He believed that some gas engineers, after the gas had passed out of the hydraulic main, allowed a certain amount of atmospheric air to be introduced. It assisted the purification. It was difficult to understand the 10 per cent. of nitrogen getting into the gas in any other fashion. How could it be understood as the result of the temporary closing of the lid of the retort? He did not know whether they had patent lids at Birmingham or those of the ordinary form. He spoke with the greatest deference on the subject, but there was the 10 per cent. of nitrogen, and those figures were quite exceptional. He certainly believed that it did assist the purification in some instances to admit a certain amount of atmospheric air. The oxygen, as shown in all the analyses had practically disappeared. What became of it? The fact of the ready absorption of oxygen in gas purification had struck him very much some years ago in the course of some works on which he was then engaged. Sulphide of calcium, as it exists in the ordinary lime purifier, was a very powerful absorbent of oxygen; and there was no cheaper or better way of getting rid of it than by such means. Sulphide of iron would, no doubt, act in a similar way. Really, therefore, the free oxygen of atmospheric air was never found in the finished gas owing to these circumstances. The report he had already alluded to would throw some light on the subject of the illuminating power of hydrocarbons. Mr. Dibdin and himself had worked it out very carefully for naphthalene, the values for which were expressed in grains per candle power per hour. The results were valuable in themselves, though he was afraid they had not been working on quite the same line as Dr. Frankland. He had no doubt that their figures, imperfect as they might be, would throw some light upon the subject. One very curious point in connection with the burning of the naphthalene, as was done in the albocarbon apparatus, was that the illuminating power, due to the naphthalene itself, worked out wonderfully closely for widely different rates of consumption of naphthalene. The figures all through the series were very concordant, though he regretted that it was not then in his power to publicly state what they were.

## Liverpool Section.

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Hon. Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be made to Edward George Ballard, Queen's Park, St. Helens.

UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL.

Wednesday, May 7th, 1884.

MR. MUSPRATT IN THE CHAIR.

## ON THE ACTION OF NITRATES UPON IRON AND SULPHIDES IN THE PRESENCE OF CAUSTIC ALKALI.

BY G. LUNGE.

THE *Journal of the Society of Chemical Industry* (1884, p. 138) contains a paper by Mr. E. W. Parnell, in



which he endeavours to refute some criticisms on a former paper of his, made in a communication contributed by Mr. J. H. Smith and myself (1883, pp. 460 and 525). Mr. Smith having left this neighbourhood, I shall answer Mr. Parnell as far as it is required to establish actual facts; but I shall abstain as much as possible from entering into a discussion about mere opinions or hypothetical explanations, quite content to leave it to the reader to decide between Mr. Parnell and ourselves. I would only point out that Mr. Parnell does not even touch upon some of the most serious objections we made to his conclusions, and confines himself only to one or two points.

In the first place, I must, however, point out that Mr. Parnell misrepresents (of course quite unintentionally, but in consequence of superficial reading) both the conditions of our experiments and the conclusions drawn from them. As for the latter, we put them as plainly as possible on p. 535. As to the former, he gratuitously assumes that we worked with crude caustic liquors—not only without any warrant for his assertion, but in opposition to our distinct statements that we made our researches with a mixture of pure materials, an indispensable condition for our investigation from its very nature. At the end of our paper (p. 532) we gave the analysis of some factory products, but quoted no experiments with the same. Consequently all Mr. Parnell's conclusions, based upon the supposition of impurity in our materials, are wrong. It is true that our solution of pure caustic soda yielded an extremely slight quantity of ammonia on boiling; but every experienced chemist knows that this is an almost inevitable occurrence; and precisely for that reason, and to render our experiments as trustworthy as possible, we expelled and estimated this nominal amount of  $\text{NH}_3$  before add-

ing the other materials; but neither iron nor sulphides were present in our caustic soda, not even in traces.

We had stated (p. 461) that on boiling a strong solution of caustic soda with metallic iron and potassium nitrate, considerable quantities of ammonia were evolved, varying according to the conditions up to a maximum corresponding to 92 per cent. of the nitrate employed. Mr. Parnell, who in his previous paper (1882, p. 135) had denied any reducing action whatever of the iron in this case, now does admit a slight action up to 3 per cent. of the nitrate, but declares our statement to be quite extraordinary—in other words, incredible. In this case either he or we must have wrongly operated. Either it is true, as we say, that metallic iron evolves considerable quantities of ammonia from a boiling solution of a nitrate in the presence of an excess of alkali, or it is not true, as Mr. Parnell implies. I shall give the reader material to form his own judgment. The experiments in question (made, like all the others, by Mr. J. H. Smith, as stated in the foot-note, p. 461) were not mentioned in detail in our paper, as this had grown to a considerable length, and we wished to cut out as many of the less important things as possible. They were given at some length in the German paper I communicated at the same time to the *Chemische Industrie*, 1883 p. 302, and I shall now quote them here as well. A solution of pure caustic soda was employed. In the experiments 1 to 8 we took 150c.c. of 90° Tw.; in No. 9, 250c.c. of 55° Tw.; in No. 10, 500c.c. of 75° Tw. This liquor was heated with the additions mentioned below, in the same apparatus as represented in the diagram, p. 461 of our paper, and with the same precautions against any liquid particles being carried over. The following results were obtained:—

Expt. No.	$\text{KNO}_3$ added.	Description of Iron added.	Weight of Iron.	Total $\text{NH}_3$ formed.	$\text{NH}_3$ formed, deducting that in original liquor.	Percentage of $\text{KNO}_3$ reduced to $\text{NH}_3$ .	Duration of Boiling.
	Grms.		Grms.	Grms.	Grms.		Minutes.
1	—	—	—	0·0027	—	—	20
2	—	clean thin wire	excess	0·0026	—	—	20
3	—	clean wire nails	excess	0·0024	—	—	25
4	—	cast-iron filings	excess	0·0028	—	—	15
5	0·5825	clean wire nails	excess	0·0226	0·0201	20·5	15
6	0·6610	clean wire nails	15	0·0495	0·0470	42·5	30
7	0·6390	cast-iron filings	?	0·1042	0·1017	91·0	20
8	0·8955	cast-iron filings	13	0·1423	0·1398	93·5	30
9	2·547	clean sheet iron	18·5	0·0148	0·0123	2·9	75
10	8·600	cast-iron ring	7·55	—	0·0164	1·15	?

These data surely bear out the conclusions we drew from them. They shew that a nominal quantity of ammonia was evolved by boiling the caustic liquor alone, that this was not increased by adding any of the descriptions of iron to be used hereafter, but that it was immensely increased by adding potassium nitrate. The last two experiments show only a slight action, evidently both on account of the much greater dilution of the liquid and of the small surface of iron employed. The cast-iron ring was weighed before and after. It lost 0·145grm. during the operation, which slightly more than corresponds to the  $\text{NH}_3$  found. In short, the experiments bear every evidence of trustworthiness.

Since, however, Mr. Parnell has thrown doubt upon this point, I have called in another witness. I caused

one of my demonstrators, Mr. Nützi, who was then entirely unacquainted with the controversy, to make a few experiments in this direction, without biasing him at all one way or another. He employed a 300c.c. flask, with a gas-delivery tube  $\frac{1}{8}$  in. wide, cut off obliquely at the bottom, the vertical part being 12 in. high, with a bulb of nearly 2 in. diameter in the middle of it. The lateral part of the tube had also an upward rise for 9 in. of its length, which arrangement certainly must have prevented anything from spurting over, more especially since the boiling liquid always remained a good deal below the bottom end of the gas-delivery tube. No aspirating apparatus was used, so that the ammonia found must have been too low. The results were as follows:—

Experiment 1: 100c.c. pure caustic soda solution

of 60° Tw., previously boiled to expel any  $\text{NH}_3$  present, 2.38grms. iron wire, 0.9425grms.  $\text{KNO}_3$ . The boiling was carried on gently, so that all the moisture was condensed in the tube and returned to the flask, and no concentration took place. After half an hour's boiling only a trace of  $\text{NH}_3$  was formed, the liquid evidently being too dilute.

Experiment 2: 150c.c. of caustic solution at 80° Tw., 14.99 grms. clean wire-nails, 0.8685 grms.  $\text{KNO}_3$ . The boiling was carried on more strongly and the vertical part of the tube kept from cooling too much, so that a concentration took place. After half an hour's boiling, 5.32 per cent.; after another three-quarters of an hour an additional 12.60; altogether 17.92 per cent. of the nitrate was recovered in the shape of  $\text{NH}_3$ .

Experiment 3: 150c.c. caustic liquor of 80° Tw., 0.6464gram.  $\text{KNO}_3$ , and 10.993grms. of very fine iron wire were boiled as in experiment 2. After three quarters of an hour's boiling 65.31 per cent. of the nitrate was recovered as ammonia, proving the efficiency of the greater surface of iron.

An accident put an end to the experiment at this point, but it was observed that ammonia was being evolved at the time. It was needless to carry the experiments further, for Mr. Nützi had already completely confirmed the results of Mr. J. H. Smith.

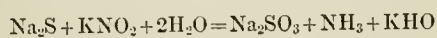
I would now beg leave to point out an oversight in Mr. Parnell's reasoning. He believes that whilst metallic iron does not reduce nitrate in the presence of alkali, iron sulphide does so to an enormous extent, acting and reacting some 300 times in one of his experiments. Since, according to his own shewing, metallic iron would reduce up to 3 per cent. of the nitrate, naturally being transformed into iron oxide, and since he (erroneously) believes our caustic liquor to have contained some sulphide, there ought to have been sufficient iron sulphides in our experiments to start that constant action and reaction assumed by him. But this assumption is entirely founded upon one experiment, recorded by him on p. 140, in which no ammonia was formed when boiling a mixture of caustic soda, sodium sulphide, and potassium nitrate, whilst ammonia appears in the following experiments, when ferrous sulphate had been added. Mr. Parnell instantly concludes that this difference is explained by the fact of  $\text{FeS}$  being formed, and being oxidised and re-formed many times over; but he does not state how much nitrate he had used in his first experiment, and as the  $\text{Na}_2\text{S}$  present was completely oxidised in the same, it is self-evident that there was at least sufficient nitrate present to perform the oxidation with formation of nitrite only, whence no ammonia could be found. In the following experiments less nitrate was employed, which necessarily caused a reduction of the same down to  $\text{NH}_3$ , and the same results would have been produced if he had left out the ferrous sulphate altogether, or else he might have added a thousandth part of what he did, and then concluded that the  $\text{FeS}$  had done its work 300,000 times over! He thinks it strange that this action and reaction should have escaped us; but I must confess to being just as little enlightened now as I was before.

Only in one point Mr. Parnell seems to be in the right against us, but in an extremely unimportant one for our controversy. We had said that the reducing action of iron upon nitrate in an alkaline solution had been made use of in a well-known analytical operation. Now it seems true that the various authors who have employed the operation in question have used iron always in conjunction with zinc. It would hence be a new observation of ours that iron alone has a similar action, of course less energetic than that of zinc, which forms

an oxide soluble in caustic soda solution, whilst iron oxide is but slightly soluble in concentrated alkaline liquors, and insoluble in dilute liquors. The last-mentioned fact may have some connection with the observation that the reducing action of iron ceases below a certain degree of concentration. The bulk of the iron oxides formed was seen floating about in the liquid, amounting to an ocular demonstration of the substantial action of iron upon alkaline nitrates, denied by Mr. Parnell.

#### DISCUSSION.

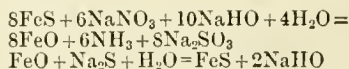
Mr. PARNELL said: In replying to the communication we have just heard, I think it advisable, first of all, to give in a few words a *résumé* of the whole question from the commencement. In a paper that I read before this Section of our Society about two years ago, I attacked the formulæ that different authorities had given as representing the reduction of alkaline nitrate to ammonia in a boiling alkaline solution. The oldest of these formulæ was published some 25 years ago, and as far as I know, up to the time when my paper was read, the formation of ammonia in this way had never been questioned. In my paper I shewed that with pure materials oxidation of sulphide by nitrate takes place without formation of ammonia, and concluded in consequence that when ammonia is produced some other element must be brought into the reaction. I also tried an experiment to ascertain whether the presence of metallic iron would cause the sulphide to reduce the iron to ammonia, but failed to find that it did so. In a paper published in our Journal in November and December last, Professor Lunge and Mr. J. H. Smith dispute my statement, and maintain that with pure materials ammonia is always to some extent produced without the presence of any other element being necessary. In criticising my former paper, and my experiments with pure materials, they remark, page 534, that I "found no ammonia is above all things remarkable." In this paper Professor Lunge and Mr. Smith first raise the purely abstract question of the reduction of nitrate to ammonia by metallic iron alone in a boiling alkaline solution. It should be clearly understood that this point is only indirectly connected with the main question under investigation, which is the reduction of nitrate by alkaline sulphide. It is quite unimportant whether the iron has a powerful or a feeble reducing action on nitrate. Admitting that some action takes place, Prof. Lunge and Mr. Smith maintain that this action going on in a solution containing in addition an alkaline sulphide "promotes" a reduction of the nitrate to ammonia by the sulphide. On page 470 they give the formula—



the nitrate having been previously reduced to nitrite by the action of the sulphide. This formula is the same that they give on page 467, as representing what takes place to some extent when no iron is present. In order clearly to understand the point at issue, bear in mind that they say that this action takes place to some extent in pure solutions, but to a much larger extent when there is concurrent action on metallic iron. In my next paper I adhere to my former conclusion, that no ammonia is formed by the reducing action of sulphide alone unless some other element is present, and that consequently all formulæ that represent such formation are incorrect. But with regard to the formation of ammonia that does take place in impure solutions, I threw an entirely new light on the matter by demonstrating that the presence of a small quantity of iron sulphide in



the liquid caused the formation of a comparatively enormous amount of ammonia. I shewed that this ammonia is produced by the reducing action of the iron sulphide on the nitrate, iron oxide being produced, which, taking sulphur from the alkaline sulphide, forms again iron sulphide, and reacts on more nitrate, this action and reaction continuing until either the nitrate or the alkaline sulphide is exhausted. It will be seen, therefore, that I dispense with the presence of iron in the metallic state, and base my explanation on its presence in the state of sulphide in the solution. In my paper I also gave an account of some experiments that led me to conclude that metallic iron has only a feeble reducing action on nitrate in a boiling alkaline solution. There are therefore three points at issue between Professor Lunge and myself: (1) The question whether pure nitrate and alkaline sulphide, in a boiling alkaline solution, in absence of any other element do or do not yield ammonia. (2) Whether the ammonia given off from impure liquors, boiling in iron vessels, is due to concurrent action of the nitrate on metallic iron, or whether it is due to iron sulphide in the solution. (3) The minor question, whether metallic iron exerts a feeble or a powerful reducing action on nitrate alone in a boiling alkaline solution. In the communication we have just heard, Professor Lunge first speaks of the last-named question. He admits that he was incorrect in speaking of a "well-known analytical method" based on this reduction. On comparing the figures now before us with the figures in my former experiments, I see that where Professor Lunge has obtained a satisfactory reduction he employed a very much larger proportion of iron, and also a very much smaller quantity of nitrate than I did. This would account in a great measure for the difference in our results. Observing the very unsatisfactory nature of some of his reductions, I think it quite possible the difference may be further accounted for by a difference in the quality of the iron we employed. Judging, however, only from Professor Lunge's figures, and bearing in mind the high temperature necessary, and also the very different action of zinc as shewn in my experiments, I think no one could maintain that my conclusion that "iron has a feeble reducing action of nitrate with formation of ammonia" can be considered unreasonable. The next point at issue is the production of ammonia from pure materials. Professor Lunge makes no reference to this in his present paper, so I think I may claim to have established the point. Professor Lunge says that he distinctly stated that he employed pure materials, but I cannot see where he describes the caustic soda he employed. I have always used caustic perfectly free from ammonia, and would certainly consider a material that gave the amount of ammonia that Professor Lunge describes utterly unfit for experiments of this nature. I cannot help suspecting that it is due to the employment of such caustic that Professor Lunge has so far failed to oxidise sulphide by nitrate without formation of ammonia. The last point at issue is the explanation of the production of ammonia in impure solutions. As before explained, Professor Lunge attributes it to concurrent action on metallic iron, illustrating it by two independent formulæ, one representing the reduction of nitrate by metallic iron, and the other the reduction of nitrate to ammonia by the alkaline sulphide. These two formulæ are in no way connected with each other, unless, indeed, Professor Lunge has become a convert to the belief in catalytic action. On the other hand, I account for the formation of ammonia in this case by the presence of iron sulphide in the solution, and maintain that the presence of iron in the metallic state is beside the point altogether. I represent the action and reaction thus:—



Now, I think there should be no doubt which explanation is the correct one. My experiments clearly shew that a mixture that before yields no ammonia, on being boiled, after the addition of a small quantity of iron oxide, gives off ammonia in abundance. In one case this amounted to 300 times the quantity due to the oxidation of the iron itself. Looking at the matter from a practical point of view, every caustic manufacturer knows that if nitre be thrown into the centre of a pot of boiling caustic liquor, an instant evolution takes place; whereas several seconds would have to elapse before the nitre could come in contact with the iron of which the pot is made. Further, on the large scale the conditions are very different to those under which Professor Lunge's experiments were made, as in the former case the nitre is added very gradually, is immediately decomposed, and the iron of the pot is practically never in contact with caustic containing an excess of nitrate. In the communication we have just heard Professor Lunge says that the action and the reaction of the iron compound was not new to him. In reply, I simply ask how this can be reconciled with his paragraph on the action of iron on page 462. He here calculates accurately the amount of ammonia due to the action on the iron, and distinctly says: "This amount, deducted from the total ammonia, was taken as being due to the reduction of the sulphide"—that is, alkaline sulphide. He represents the iron as acting once, and once only. Further, what is most perfectly clear and conclusive, he says in the same paragraph: "The oxidation of the sulphide of iron and its conversion into ferrous hydrate has no connection with the hypothesis." How could he be alive to the action and reaction and say this? Although in many of his experiments iron sulphide must have been present, in no single instance does he connect it in any way with the formation of ammonia, or even hint at action and reaction. In conclusion, I can only follow Professor Lunge's example and leave it to those who are interested in the matter to judge for themselves from the evidence before them.

#### \* REPLY.\*

Professor LUNGE: In claiming an author's right to reply to the criticisms passed on his paper in discussion, I shall endeavour to put the points clearly and concisely. In the paper published jointly by Mr. J. H. Smith and myself, we had refuted well-nigh all the conclusions drawn by Mr. Parnell in his first paper. In his second paper, Mr. Parnell takes up only one or two of the contested points. He still denies a substantial reducing action of iron on nitrate in boiling alkaline solutions, and he ascribes our results, above all the evolution of ammonia, to our having used "crude liquors," containing sodium sulphide, and hence iron sulphide. Both these points are refuted in the present paper. I have proved a substantial reduction of nitrate by all descriptions of iron, and I have given a full explanation of what Mr. Parnell calls "the very unsatisfactory nature of some of our reductions." I have further stated that our caustic soda contained no trace of either iron or sulphide; it was the purest caustic soda of trade, but, like every other description of caustic I have met with, yielded a trace of ammonia on heating. Every analytical chemist who has had occasion to go into this matter, *e.g.* in per-

\* Professor Lunge's paper having been received as a communication to the Journal, and no column for "Communications" now existing, it was read as a paper before the Liverpool Section, proof, with discussion, under these circumstances, being forwarded to the Author. He sent the above reply.

forming Wanklyn and Chapman's water test, knows that the above circumstances must be taken into account. Mr. Parnell now says that he always used caustic soda perfectly free from ammonia; but in describing his experiments, he did not say a word about having tested for it. We employed, as stated, 150c.c. caustic soda solution of 90° Tw., which means 90 grms. NaOH; this yielded 0.0024 to 0.0028 grms.  $\text{NH}_3$ , or 3 parts for 100,000 NaOH. Mr. Parnell considers such soda to be "utterly unfit for experiments of this nature;" he consequently claims to have "established the point" of the production of ammonia from pure materials, forgetting that a negative can never prove a positive in this manner. But, in any case, how could that trace of ammonia, given off by our caustic, influence our results as to the action of nitrate on  $\text{Na}_2\text{S}$ ? His conclusion may be formulated thus: "I contend that ammonia is not produced by the action of nitrate upon sulphide in pure caustic solutions, but only in the presence of iron sulphide. Lunge contends the contrary; but all his experiments are valueless, because his caustic soda, although perfectly free from iron, yielded 0.003 per cent. of ammonia [always accounted for!]; it was therefore 'impure,' and utterly unfit for proving that iron has no action in this matter."

This is Mr. Parnell's argument.

I must, however, again point out that Mr. Parnell has not proved that "a mixture that before yields no ammonia, on being boiled, after the addition of a small quantity of iron oxide, gives off  $\text{NH}_3$  in abundance." I have shewn that Mr. Parnell, who, according to his own statement (p. 138), did not employ the identical mixture in both experiments, in his first experiment must have employed such an excess of nitrate, that only nitrate was produced, but no ammonia could be found. It is also useless to argue that on the large scale the iron acts less than in our experiments. This is really quite beside the mark, for our conclusion (vol. ii. p. 535) literally ran thus: "The presence of iron in every case hastens, but does not alter, the results of the action. In conclusion, Mr. Parnell has strangely misunderstood an expression of mine. I had conclusively shewn that the ferrous sulphate used by Mr. Parnell had nothing to do with his results, and I endeavoured to reduce this *ad absurdum* by multiplying his 300 actions up to 300,000. I said, moreover, in the same breath: "He might have left out the ferrous sulphate altogether." From this, Mr. Parnell infers that I claim having recognised, before him, the repeated action and re-action of the iron sulphide! I really do not claim this, but willingly leave any credit there is in it to Mr. Parnell. I distinctly beg, in conclusion, to state my conviction, that Mr. Parnell has not succeeded in disproving a tittle of our objections, raised against the conclusions of either his first or second paper.

## Manchester Section.

Chairman: Ivan Levinstein.

Vice-Chairman: George E. Davis, F.I.C., F.C.S.

### Committee:

F. B. Bengier.  
R. F. Carpenter.  
C. Estcourt.  
H. Grimshaw.  
Peter Hart.  
J. von Hohenhausen.

T. Jackson.  
S. Mellor.  
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R. Angus Smith.  
W. Vickers.  
D. Watson.

### Hon. Sec.:

J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester

## MANCHESTER TECHNICAL SCHOOL,

June 10th, 1884.

The next Meeting will be held as above at Half-past Seven in the Evening, when the following communications will be made:—

Mr. WATSON SMITH—"A Rapid Method for Estimating Caustic Alkalies in presence of Alkaline Carbonates."  
Mr. G. E. DAVIS—"A Note on the 'Extraction of Benzene from Coal Gas.'"

The SECRETARY—"A New Process for the Manufacture of White Lead."

The CHAIRMAN—"A few Remarks on 'Paraxylene.'"  
Discussion of the Report of the Committee on "Standard Methods of Sampling, Analysis, and Stating the results."

## ON THE OXIDATION OF CELLULOSE.

### PART II.

BY C. F. CROSS AND E. J. BEVAN.

THE CHAIRMAN, in opening the proceedings, gave a short recapitulation of Messrs. Cross and Bevan's last paper, and he then said, that however interesting the conversion of cellulose into oxycellulose may be to the technologist and chemist—as far as bleachers are concerned, it is a process which must be carefully avoided, as by chlorinating or oxidising the cellulose the material will be weakened. What the bleachers of cotton and other goods desire is to obtain a clear bright white, without injuring or weakening, as far as possible, the strength of the material. Mr. Thompson claims by his process the saving of chlorine and retaining the fabric in a better and stronger condition than by employing the usual process. He does bleach with minimum quantities of chlorine, using as an assistant carbonic acid gas. We have already heard from Messrs. Cross and Bevan, that Witz has proved that carbonic acid increases the action of chlorine in bleaching. I will now call, with your permission, on Mr. Thompson to fully explain his process, and the working model, which he will be good enough to exhibit and explain to you this evening; but I desire it to be clearly understood that our Society does not lend itself to advertise new processes. We thought, however, in the case of Mr. Thompson that, as his process has been recommended by some and has been favourably reported upon by many, it would be of advantage to our members—especially as his process, as far as chemistry is concerned, is plausible—to give Mr. Thompson an opportunity of explaining it. We have this evening about 500 gentlemen present—of whom I notice a large number are bleachers, printers, paper makers—gentlemen who are all more or less practically acquainted with the process of bleaching, and we may safely leave it in their hands to get in the discussion all the information from Mr. Thompson which is necessary to form a correct opinion of the usefulness of his process.

Mr. THOMPSON then proceeded to explain his bleaching kier, after which Messrs. Cross and Bevan read their supplementary paper.

Mr. Cross then said: We wish to supplement our last communication to the Society by a few observations on certain of the points therein raised. I am very much afraid that what we brought before you at the last meeting as incidental merely to the more general account of the chemical activity of cellulose, has, for reasons best known to yourselves, become magnified into the chief feature of attraction for the majority of those present this evening. We promised to demonstrate to you the Thompson bleaching proofs, and we have the appliances here for the purpose. With the more practical side of the questions which it raises we shall not need to concern ourselves further; we leave them in



more experienced hands. At the same time we would express our opinion that the processes of bleaching at present practised in this district are capable of improvement; some of the directions in which improvement is called for appear to us to be considered in this particular process; more especially, however, speaking as it is our object to do from the general aspect of these questions, we lay stress upon the results of M. Witz as affording new and valuable criteria of the efficiency and mode of action of this and other bleaching processes. We may also add that, of course, it is not pretended for the Thompson process that it offers any other novelty than in the mode of applying principles familiar to all—*non nova sed nove*. Before now proceeding to the demonstration of this process we venture to draw your attention to a few observations by which we desire to supplement our last paper.

*a. Oxycellulose.*—It may be doubted whether the affinity manifested by this compound, *i.e.*, by cotton after being subjected to the action of bleaching powder and other oxidising agents is truly a chemical affinity in the strict acceptance of the term, for the chemical change or alteration in composition which cellulose undergoes when acted upon by oxygen, is accompanied by a structural modification more or less profound, sufficient, according to the generally accepted theory of dyeing, to account for the altered relationship of the fibre to colouring matters. Further, as we have stated, the combination of the oxidised fibre with colouring matter is of so weak an order as to be overcome by the action of water, and this fact might be taken as shewing that the union in question is rather of a mechanical than a chemical nature.

The only modern investigation of the theoretical basis of dyeing with which we are acquainted is that published by Crum in 1863 (*Chem. Soc. Journ.* 16, 1 and 404). His repudiation of the chemical theory of dyeing advanced by Macquer, Berthollet, and others, is very emphatically expressed in the words "no one who is at all conversant with the rationale of the processes of dyeing, and with the laws of chemical combination as they are now understood, can examine the question without arriving at an opposite conclusion" or theory).

Twenty years have elapsed since these words were written, and the progress of our knowledge of the structure and composition both of the organic colouring matters and of the fibres which they are employed to dye, will undoubtedly enforce a considerable modification of the view therein expressed. It would carry us far beyond the scope of this paper, to attempt a thorough discussion of so wide a subject; and we must content ourselves with stating one or two considerations upon the special point which has been raised. It has been shewn that oxidised cotton or oxycellulose is a chemically active substance; it reduces cupric oxide when boiled with the solution of the sodio cupric tartrate, and the resulting cuprous oxide is deposited upon the fibre in a state of such intimate union that it gives the effect of an orange dye. It has a remarkable selective attraction for vanadium, removing this element from solutions containing almost inconceivably minute traces of its soluble compounds. But perhaps the most obviously chemical feature of its combining activity, is manifested in its union with certain colouring matters belonging to a well-marked group, *viz.*: the basic colouring matters, together with its repulsion of certain others, belonging to the group of acid colouring matters. We fail to see how this could be accounted for otherwise than by referring the dyeing capacity of the fibre to its chemical constitution. It is also worthy of notice that in the case of certain of the cottons investigated by Crum he noticed a marked difference in its

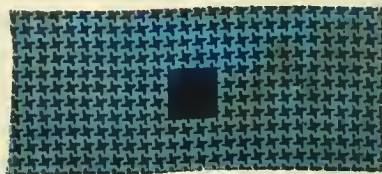
dyeing capacity—for alizarin—before and after bleaching, the bleaching appearing, from his coloured plates, to have altogether destroyed the power of taking up this colour. This result he likewise refers to physical causes. With regard to Crum's researches we would point out that this Turkey red dyeing selected by him as the case for special investigation, was an unfortunate choice, resting as it does upon the indirect method or methods of mordants, of which, moreover, it is not a typical instance; and, also, in being calculated to confirm a firm view of dyeing as a phenomenon controlled by *mechanical* rather than *chemical* laws, under the influence of which he appears to have worked. In further investigations of the subject it will be well to confine ourselves as far as possible to *a posteriori* conclusions, and to avoid inconsiderate use of mere terms, for, it cannot be doubted that in the phenomena of dyeing we have in operation a highly complex set of conditions to deal with, which it may be found impossible arbitrarily to classify into physical and chemical.

In developing his views Crum has recourse to considerations of surface, porosity, capillarity, in other words to the external or visible configuration of the fibre substance, as the main factor in its dyeing capacity. That he over-estimates the influence of these may be readily shewn by reference to the methods of the microscopist. The well-known operations of staining sections and objects for examination by means of the microscope, afford abundant evidence that the most refined methods of visual analysis fail to detect the cause of the attraction of colouring matters from their solutions. Of two cells lying side by side similar in point of structure and even function, one freely attracts a certain colouring matter, the other not at all. This is one out of numerous examples which might be cited to shew that dyeing capacity is the expression of attractions different from those usually included under the term physical. One more instance we may mention. The flax and jute fibres are very similar in structure and identical in their functions in the plant, both being fast fibres; of these, jute has a powerful attraction for a large number of colouring matters, flax on the other hand, so little, that it can only be dyed by indirect methods. This great difference is accompanied by an equally great difference in chemical composition and constitution, and we cannot avoid the conclusion that these facts are essentially correlated, *i.e.*, that the difference in dyeing capacity is primarily the expression of the difference in chemical composition.

At present we do not feel justified in exceeding the limits of this very superficial discussion of a deep problem which will doubtless continue to divide theorists.

As a matter of practical science we think sufficient ground has been shewn for investigating the dyeing capacity of the cellulose fibres, and the phenomena of dyeing generally from the point of view of chemical constitution. And the same will hold for printing

#### ILLUSTRATION.



Cloth converted into oxycellulose and printed—Methylene Blue. (Kindly supplied by M. Georges Witz, of Rouen.)

also, at least in the majority of styles. The chemical activity of these fibres indeed, we wish to see clearly recognised as a basal principle of the textile arts:



scouring, bleaching, dyeing, and printing, we especially desire to see established upon fundamental chemical principles, and it is for this reason that we have thought proper to reproduce to the Society the results of M. Witz and others, who have laboured in this field of research.

*The Thompson Bleaching Process.*—We drew attention to this process as involving the practical application of certain of the results established in M. Witz's researches. It is perhaps scarcely necessary to point out that this connection is one for which we are responsible, and that in so far as regards their origin and development, the two matters are entirely independent of one another. In reference to this bleaching process, there are one or two points which we desire to add.

The loss of weight sustained by cotton and linen goods treated by this process has been found to be extremely small, viz., excluding the sizing materials, 4.5 per cent. in the case of cotton, 10.12 in that of linens.

This may be due to two causes—(1) mechanical, (2) chemical. (1) The handling of the goods is reduced to a minimum. (2) The chemical action of the bleaching powder upon the fibre constituents is minimised (*a*) by reason of its being supplied in such small quantities relatively to the weight of the goods as to confine its action, as much as possible, to the more easily oxidisable, *i.e.*, the non-cellulose constituents of the fibre, and probably also (*b*) by reason of the presence of carbonic anhydride under pressure, which would oppose any action of the bleaching powder upon the cellulosic constituents of the fibre tending to produce this gas; this probable effect being deduced from the well-known law, that a reaction is modified or arrested by antagonising any of the physical changes incidental to the reaction. In both directions, therefore, this process tends to conserve the fibre.

There are certain minor advantages which we might also have noticed, chiefly those which contribute to the uniformity of the bleach. The evenness of the bleaching is a consequence partly of the method of supplying the bleaching agent in limited quantities, partly the circulation of liquid which the process involves. The tendency of those modifications to produce uniformity of result is obvious. As to these the conduct of the operations in a closed vessel, *i.e.*, with exclusion of air and light, and the condition of this bleaching process will be found to conform to those deduced from the "Researches of M. Witz" as productive of the best results.

The CHAIRMAN: We have now heard the very interesting supplementary paper of Messrs. Cross and Bevan, and the greatest credit is due to them and Dr. Witz for the great trouble which they have taken, not alone in clearing up some mystery, which was always attached in cases of deficient bleaching, but of having opened an entirely new field of investigations to the technical chemist and colourist. The specimens of prints which have been shewn to us, and have been produced by printing on cloth, first an oxidising mordant with the object of converting oxycellulose into cellulose, and then passing the goods through a basic aniline colour, are exceedingly pretty, and may probably add a new style to the art of calico printing. In the following discussion I would like to call your attention to the fact that if there is a practical process of converting the entire cellulose of the cotton into oxycellulose without injuring the strength of the material, and if the colours produced by dyeing oxycellulose with certain aniline dyes are faster than those produced by dyeing ordinary cotton with the same colours, and, further, if the oxycellulose resists as well alkalis as the cellulose, that oxycellulose must be

destined to take a prominent part in the arts of dyeing and printing. If I understood Mr. Cross aright, he stated that basic colours and oxycellulose combine.

Mr. CROSS: Yes.

The CHAIRMAN: Is this combination a chemical combination?

Mr. CROSS: I cannot answer this question.

The CHAIRMAN: I understood from the paper that the combination was a chemical one, but I suppose I was mistaken. It might occur to you, that the greater affinity which colouring matters and metallic mordants possess to oxycellulose is, probably, more due to a change in the structure which the cellulose undergoes in the oxidising process, than to a true chemical combination between the two. I shall now be much pleased to hear your opinions on Mr. Thompson's process and new bleaching kier, of which he has shewn us his very neat working model, and also on Messrs. Bevan and Cross's most interesting and valuable two papers.

Mr. MELLOR: Is the cloth made tender in the have process of the formation of oxycellulose? I now before me one of the samples sent round for inspection, printed with small traces of vanadium, and I must say that it is very tender. In the light expressed by the Chairman, if we have the prospect of a new valuable material, it is of the utmost importance that we should have some exact information on the matter.

Mr. DAVIS: I should like to ask Mr. Thompson whether the fibre was tendered in any way by the deposition of carbonate of lime? Some time ago a question was raised by some German chemists as to the tendering of some serviettes at an hotel, and it was distinctly proved that it was owing to the serviettes having been dipped in lime water that caused a considerable tendering of the fibre, so that when they were pressed they generally cracked along the folds, and it seems to me that the carbonic acid gas might have the effect of tendering indirectly the fibre in this way. Mr. Thompson's process seems a most admirable one, because the cloth being folded up and not touched until the operation is complete makes it very valuable. I am much surprised to find the white developing so fast, and we shall very soon see if goods can be bleached in so short a time.

Mr. HOWELL: May I ask Mr. Thompson if his process will preserve the headings, as they are almost as valuable as the cloth itself. I should like to know if the piece in the model has been limed or ashed? If it has been treated with alkali, of course a great part of the work is already done.

Mr. THOMPSON: It has been boiled in caustic soda.

Mr. CUNLIFFE: May I ask Mr. Cross's opinion as to the formation of oxycellulose by the Thompson process. I think Mr. Cross shewed us some cloths that were exposed to the action of carbonic acid gas, and described the action of the carbonic acid gas and the atmosphere during bleaching. Now, I thought there would be a considerable quantity of this carbonic acid gas present during his bleaching process, and I should like Mr. Thompson to demonstrate to us the financial advantages. I understand that he claims a very great reduction of cost by his process. I would also ask him if there is any advantage in using carbonic acid instead of hydrochloric acid, and what is the cost of the material. I presume that in scouring the cloth liming is essential. We use that in printing because it is a cheap alkaline, and removes many of the injurious matters. I don't see how his process applies to printers.

Mr. FLETCHER: Is Mr. Thompson's bleach weaker after he has used it, and does he wash the cloth after passing it through the chloride of lime?

Mr. THOMPSON: I wash it in cold water.



Mr. FLETCHER : I understood Mr. Cross to say when the cloth is printed on the bleach was not required to be so white. If you want a bright colour, you must print on a good white ground. Will Mr. Thompson's process bleach oil waste or rags used by the Railway Companies?

Mr. HILTON : Does Mr. Thompson claim any superiority by this machine of his over the Masen-vacuum process of bleaching? Mr. Mason, as far as I remember, declines to use his pump for the purpose of pumping liquids. He simply exhausts, by means of an air pump, his reservoir, and thus the atmospheric pressure drives the bleaching agent into the reservoir. It seems to me that Mason's process would be the most economical.

Mr. KNOWLES : It is no news to the majority of those present that there are two sorts of bleach—madder bleach and printer's bleach. Some time ago, I obtained a few samples which had been bleached by the Thompson process, and I have them here. I have also a fent taken from the best madder bleach in our own kiers, and also a fent of Mr. Thompson's bleach by the process now before you, and another fent which was first boiled in caustic soda and then passed through his new process, and also another which was boiled in cyanide of potash, which he also recommends. As a matter of cost, I don't know how that will come out, but these fents were all stitched together and run through a mordant. In one case you can certainly see a slight difference; it shews oxycellulose. This is madder bleach out of our own kiers. This passed through Mr. Thompson's kier without previous boiling. Before this was done, we could not tell one from the other. As far as I am able to say, unless some improvement has been made since in printer's bleach, which will shew a good white, I cannot see that it is a success. If we turned out goods like that I don't think our merchants would receive them. Of course, that which was boiled in caustic soda was better than the others.

Mr. BARKER : By the Thompson process of bleaching, is the cloth what is technically called "got up" in the finishing; and also if a large quantity of cloth is put in the kier, will the fluid penetrate evenly?

Mr. GRIMSHAW : It seems to me something extraordinary that we should have three papers in succession on a similar subject, that of dyeing and bleaching. I allude first of all to Mr. Sharp's paper on the different modes of preparing and bleaching for dyeing in Lancashire, as compared with Yorkshire, in which he attributed the difference in the colours to the action of chloride of lime, which caused the cloth to take the dye much better. Then we had at our last meeting Messrs. Cross and Bevan's important paper, in which they laid before us the extraordinary modification which cotton cloth undergoes by being converted into oxycellulose; and it seems to me that we are almost on the track of something which will have an important bearing on the bleaching of cotton goods, because it does certainly appear that this formation of oxycellulose, when cotton is subjected to the oxydising action of chlorine, which for years back has puzzled dyers and bleachers, would seem to be almost explained at one sweep by the formation of oxycellulose on the cloth. There is one question which Mr. Thompson would do well to answer, and that is the way in which he successively treats the cloth with chlorine liquor, and then with carbonic acid gas. Has he succeeded in causing the two liquids to penetrate the cloth evenly throughout? I think that is one of the most important practical problems which will have to be overcome.

Mr. NORTON : I have been in correspondence with

a gentleman at Cambridge, who has examined fibres under the microscope, and he informs me that nearly all cellular tissue had pits in the cell walls, and this accounts to some degree, no doubt, for the irregularity in the distribution of the colours upon the dyed goods.

Mr. Cross : The question of the formation of oxycellulose in Thompson's, as compared with the ordinary process, has not yet been solved; by anticipation there would be less tendency to oxydize the cellulose, and this was confirmed by the samples of cloth bleached by this process, which had just been exhibited to the meeting by Mr. Knowles.

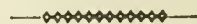
Ordinary bleached calico gave evidence of slight oxidation to oxycellulose, but there was not a marked difference in its attractions for colouring matters belonging to the two groups of acids and bases. In the fully oxydized calico on the other hand, so great was this difference that, when compared with ordinary bleached calico, its behaviour to the acid colouring matters could only be described as a repulsion, whereas the attraction for the base colouring matters was much intensified.

In reply to a number of questions propounded by Messrs. Mellor, Davis, Knowles, Grimshaw, Hunter, and others, Mr. Cross gave the following additional particulars:—Mr. Witz has succeeded in printing by means of oxycellulose, without any sensible tendering of the cloth.

The question of the application of the Thompson bleaching process to printing and dyeing, had yet to be investigated; so far the process has been worked only in regard to market bleaching. From a three years' experience of the working of the Mason vacuum kiers, he thought the Thompson method quite as efficient in point of even distribution of the bleaching liquor, and its permeation to the centre of heavy makes of goods.

The question of the formation of oxycellulose in this, as compared with the ordinary processes, had not yet been solved; by anticipation there would be less tendency to oxydise the cellulose, and this was confirmed by the samples of cloth bleached by this process, which had just been exhibited to the meeting by Mr. Knowles. Ordinary bleached calico gave evidence of slight oxidation to oxycellulose, but there was not a marked difference in its attractions for colouring matters belonging to the two groups of acids and bases. In the following oxydised calico, on the other hand, so great was this difference, that when compared with ordinary bleached calico its behaviour to the acid colouring matters could only be described as a repulsion, whereas the attraction for the basic colouring matters was much intensified.

In regard to the preliminary process of cleansing or "bowking," the choice of a detergent would be determined by the considerations which usually guide the bleacher. So also any subsequent process incidental to "finishing," or preparation for printing. The process has been considered only in reference to its bearings upon the chemistry of cellulose. The estimated cost of the process was based upon the present mode of evolving the carbonic acid, viz., HCl and CaCO<sub>3</sub>. The application of a more economical method was under consideration.



## THE VALUATION OF TARTAR EMETIC

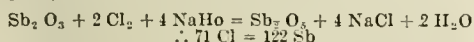
BY W. B. HART.

THE adulteration and consequent lessening of the valuable ingredient in drugs and dye-ware tends to develop methods of analysis, by which the commercial value of them may be rapidly determined,

and thus the user and honest manufacturer be protected. Tartar emetic, whose value to the dyer depends solely on the amount of antimony it contains, has of late been lowered in quality, until in some cases it contains only about one-half the amount of metal that a good commercial sample should have. The usual method of estimating the antimony in this salt is by means of a standard solution of iodine, as recommended in most volumetrical analysis manuals. This method gives good results in careful and patient hands, but I find that it can be well replaced by a solution of calcic hypochlorite, or common bleach liquor, of a strength of about 2° Twaddell. The value of the hypochlorite can be found by using a standard solution of sodium arsenite. The sodium arsenite is the usual decinormal solution, made by dissolving 4.95grms. of pure arsenious acid in a solution of sodium carbonate, and when cool diluting to 1 litre. About 25grms. of sodium carbonate are required.

1c.c. = .00355grms. of Cl, or .0061grms. Sb.

The usual potassic iodide and starch paper are needed. The process is conducted as follows:—A weighed portion of the tartar emetic is dissolved, assisted by heat, cooled, and made alkaline with sodium carbonate. A known amount of the calcium hypochlorite solution is added in excess, this being shewn by the blue colour which a drop of the liquid gives to the starch paper. The excess of calcium hypochlorite is now found by titrating with the standard sodium arsenite, until the liquid ceases to give the blue colour to the starch paper. The value of the hypochlorite added is found by taking an amount equal to that put to the tartar emetic, or an aliquot portion may be taken, making alkaline as before and titrating with standard arsenite, Penot's method. If an aliquot part is taken, the value of the whole is then to be calculated. The worth of the total hypochlorite added being known, and that of the excess also known, the amount of hypochlorite and therefore chlorine used to oxidize the antimony is thus obtained by difference. This also gives the amount of antimony. The reaction that takes place is as follows:—



This process is rapid and, for all practical purposes, accurate the end of the reaction being sharp, and denoted at once, which with the iodine process is both tardy and tiresome.

The calcic hypochlorite solution, the value of which should be found at least once a day when required, must be kept in a stoppered bottle in a dark place, as it decomposes quickly if exposed to light and air. Even then it will not be of use long, but a fresh solution can easily be made.

The following are the results obtained by this process, along with those obtained by the iodine method, shewing comparison :

No. 1.	Mean of 3 trials.	Iodine Process.	Chlorine Process.*
		37.41 % Sb	37.29 % Sb
No. 2.		37.048 % Sb	36.914 % Sb
		36.896    ..	36.820    ..
		37.000    ..	37.020    ..
	Mean	36.981 % Sb	36.928 % Sb

This method might be reduced to a comparative test only.

Let a standard sample of tartar emetic be procured in which the amount of antimony is known. Equal weights of the standard sample and of the sample to be tried, are dissolved separately. From a Bink's or glass tap burette is added to the standard sample a solution of bleach liquor until the liquid just blues the aforesaid starch papers. The amount of bleach

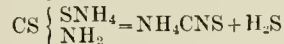
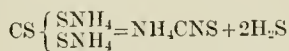
liquor used is noted. The second sample is treated in the same way. The relation of the antimony in the samples will be in the direct ratio of the volume of bleach liquor taken, and therefore, in dyeing the amounts to be taken to produce a certain shade will be in the inverse ratio.

*Example.*—Equal weights of the standard sample (1), and of the second sample (2), were dissolved. No. 1 took 30 volumes of bleach liquor, and No. 2 took 20 volumes; then the ratio of the amount of antimony is as 3 to 2. Therefore, in practical work, for every 2 parts of the standard sample, 3 parts of the sample in question must be used and those give the same result.

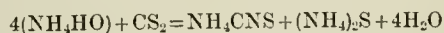
## ON THE ESTIMATION OF CARBON DISULPHIDE IN BENZOLS AND CRUDE NAPHTHAS.

BY PHILIP HOLLAND AND HARCOURT PHILLIPS.

THE presence of carbon disulphide in benzol is of serious importance both to the consumer and to the manufacturer—to the former as being hurtful in some processes to which a benzol is to be applied, and to the latter as representing an article, unless special care is taken in making the test, to appear richer in real benzene than is actually the case. Some little time ago we were requested to examine a number of samples of crude benzol for carbon disulphide, and though we were then adopting the well-known method of Mr. B. Nickels, it seemed to us desirable to have an alternative one. Some of the work done in this direction forms the subject of the present paper. Though several plans were tried of so treating mixtures of pure carbon disulphide and benzene as to get the sulphur in a form suitable for weighing, it was not until considerable time had been expended, perhaps not altogether wasted, that we decided on a process which has given satisfaction. Before describing the method we will refer to an observation made first, we believe, by M. Gélis (*Moniteur Scientifique*, 1861, also *Brochure Soumise au jury international de 1862*), viz., that carbon disulphide when digested with aqueous ammonia, yields ammonium thiocyanate. We mention this reaction as we hoped to found a quantitative method for the estimation of carbon disulphide upon it. Millon considers the reaction to consist in the formation of ammonium thiocarbonate and thiocarbamate, which, when their solution is boiled, yield ammonium thiocyanate with elimination of sulphuretted hydrogen. Thus—



Reduced to a simple expression, this reaction may be represented thus :—



(Emerson Reynolds, *British Journal of Photography*, 1863.) It may not be out of place here to mention that Gelis proposed to use this reaction as a point of departure in the manufacture of potassium ferrocyanide. Though his scheme was ingeniously based on several distinct reactions not requiring high temperature or costly apparatus, he does not seem to have been able to supplant the time-honoured but very wasteful prussiate process. Nevertheless, he has indicated a plan of approximately linking carbon to nitrogen by the interaction of two such easily-obtainable bodies as



gas liquor and carbon disulphide. The reason the Gélis process proved unprofitable was owing, we are informed, to the very serious loss of ammonia it entailed. We, however, are inclined to the opinion that this was not the only loss sustained, for 48 experiments, carefully conducted by ourselves, failed to give us more than from 60 to 70 per cent. of carbon disulphide converted into ammonium thiocyanate. In one or two instances we did indeed obtain 90 per cent. of decomposition, but on a repetition by precisely the same method we at times only got perhaps 60 per cent. What is the cause of this incomplete reaction we are not able to say, still it is interesting in this connection to learn that Schultze (*Journal für prakt. Chem.* 27, 578) obtained from 350grms. of carbon disulphide when digested with alcoholic ammonia, 280grms. of dry ammonium thiocyanate, or 80 per cent. of decomposition. Our experiments were conducted in sealed tubes, with varying amounts of alcoholic ammonia, etc., and the heating carried to different temperatures for different lengths of time. The ammonium thiocyanate so produced was estimated as cuprous salt.

It will probably be sometime ere the reaction between carbon disulphide and ammonia will cease altogether to allure manufacturers. As an instance in point, we may remark that a patent has recently been secured which provides for the simultaneous production of ammonium thiocyanate and reduction of nitrobenzene by means of the ammonium sulphide formed. We understand that two or more vessels are employed, and so arranged that the ammonium sulphide can be driven from the first into a second containing nitrobenzene, which becomes reduced with separation of sulphur according to Zinin's well-known reaction. So much, then, for the thiocyanate process, and our failure to found upon it a reliable method for the estimation of carbon disulphide in benzols. We shall now describe one, in part suggested by the previous experiments, and which has given good results when tried on benzols containing known amounts of carbon disulphide. We may remark here that special means were adopted, and care taken to free all the reagents used from sulphur compounds both free and fixed. The process shortly consists in heating the benzol to be tested with ammonia and a metallic oxide in a sealed tube. By this means the sulphur of the ammonium sulphide formed is fixed as a metallic sulphide, and ultimately oxidised along with that of the thiocyanate, and weighed as barium sulphate. The apparatus and chemicals required are: Pieces of combustion tube about 12in. or 13in. long, sealed at one end, and drawn out at the other extremity so as to form a funnel; an accurately graduated pipette divided into tenths of a cubic centimetre; a solution of ferric chloride, containing 240grms.  $\text{Fe}_2\text{Cl}_6$  in 1,000c.c.; and strong ammonia.

The estimation is carried out as follows: Into a tube is placed 2c.c. of the benzol to be tested, next 5c.c. of the ferric chloride solution, and finally 10c.c. of ammonia. The tube is now carefully sealed and well shaken. It is then wrapped in a cloth, and immersed in boiling water for an hour or so. At the expiration of that time, the tube is taken out, allowed to cool, and opened. The contents are transferred to a flask holding about 16oz., and evaporated just to dryness. This may be done over a Bunsen flame with careful management. 20c.c. of fuming nitric acid are now added all at once to the residue in the flask, and the whole is boiled nearly to dryness. As a rule the sulphur will be found to have been completely oxidised. Should this not be the case, however, a little more nitric acid will generally effect it. Hydrochloric acid is finally added, and some water, and the solution filtered. The sulphuric acid in the filtrate is then determined as barium sulphate in the

usual manner. We should mention that as it is not possible to get the whole of the ferrous sulphide, etc., out of the tube with water alone, this is best effected by means of a little hydrochloric acid to which is added a few crystals of potassium chlorate.

We now come to the application of this process to commercial benzols. It was, however, necessary first of all to ascertain with what degree of accuracy carbon disulphide could be estimated when mixed with benzene in known proportion. For this purpose the carbon disulphide was weighed out in a stout glass bulb, which was placed in a tube with a small piece of glass rod. 2c.c. of pure benzene were next added, along with ferric chloride and ammonia, and the tube sealed. By careful shaking, the carbon disulphide was liberated from the bulb, and after digestion and oxidation was estimated in the manner just described. The weight of carbon disulphide taken in these test experiments has been calculated into volume, and the following numbers express the percentage by volume of carbon disulphide found in the mixtures so made up:—

Test Mixture made up contained by Volume.		Found
	% $\text{CS}_2$	% $\text{CS}_2$
1. ....	1.65	1.63
2. ....	2.81	2.71
3. ....	3.3	3.24
4. ....	4.5	4.26
5. ....	4.83	4.77
6. ....	5.04	4.90
7. ....	5.0	4.78
8. ....	5.0	5.03
9. ....	5.0	5.33

These numbers being deemed satisfactory, we next turned our attention to the determination of the carbon disulphide in our commercial samples, and we may remark in passing that 2c.c. or 3c.c. are amply sufficient for each estimation.

The following are a few of the numbers obtained:—

#### ORDINARY LANCASHIRE TAR BENZOLS.

	% $\text{CS}_2$	%	%
50 per cent. ... (a) 1.165 ... (b) 0.825 ... (c) 0.894			
90 per cent. ... (a) 1.625 ... (b) 1.975 ... (c) 1.930			
Toluol per cent. (a) 0.161 ... (b) 0.170			

#### CARBONISING BENZOLS.

	% $\text{CS}_2$
(a) .....	0.178
(b) .....	0.131
(c) .....	0.174
(d) .....	2.450
(e) .....	0.134
(f) .....	0.246

#### ORDINARY CRUDE NAPHTHAS.

	% $\text{CS}_2$
(a) .....	0.143
(b) .....	0.207

#### SPECIALLY PURIFIED BENZOLS.

	% $\text{CS}_2$
50 per cent. ....	0.192
90 per cent. ....	0.204

The following were "pure benzols" procured from various dealers in chemicals and chemical apparatus:

	% $\text{CS}_2$
(a) .....	0.716
(b) .....	0.551
(c) .....	0.722
(d) .....	0.684

Other plans of oxidising the ferrous sulphide may, of course, be adopted, such as the use of potassium chlorate with hydrochloric or nitric acids. Or the contents of the tube may be transferred to a platinum dish, dried down, and fused with potassium nitrate and sodium carbonate. In this case, however, it is better to substitute a hydrated oxide, such as hydrated oxide of iron or zinc, for the ferric chloride solution, on account of the spurting produced by the liberation of ammonia from the ammonium chloride formed from the ferric chloride. In one set of entirely different experiments, when using hydrated oxide of lead to retain the sulphur, it suggested itself to us that

we could, perhaps, take advantage of the gradations of shade observable in the sulphide of lead, and so work out a colorimetric process for estimating the sulphur compounds in crude benzols. When opportunity offers we intend experimenting in this direction.

In conclusion, we would anticipate an objection which may be raised to the method we have set forth: viz., that it may return any other sulphur compounds present in a benzol, as carbon disulphide. To this we would reply that these bodies are probably only present in very small quantity, and are, moreover, impurities in the benzol. We do not claim for our process that it discriminates between these different sulphuretted bodies, but trust that it may be looked upon simply as a convenient commercial method for the determination of the sulphur compounds generally in benzols and naphthas.

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## ON ALGIN.

BY ED. C. C. STANFORD.

THE shores of our British Isles are surrounded by a broad fringe of aquatic vegetation known as the marine algæ. Most of these grow on that debatable flotsam and jetsum ground between high and low-water mark, others grow only below low-water mark. On all rocky shores there are certain well-defined varieties, which grow in a regular order. We find first the *Fuci* or black wrack, all partially submerged. That nearest the shore, and therefore the least submerged, is the *Fucus vesiculosus*, or common bladder wrack, with a double line of air vesicles; then follows the *Fucus nodosus*, or knobbed wrack, with a single line of air vesicles; and lastly, the *Fucus serratus*, or black wrack, with no air vesicles. Then, completely submerged, are the *Laminariæ* or red weeds—the *Laminaria stenophylla*, with a short thin stem and long frond, and the *Laminaria digitata*, or deep-sea tangle, with long thick stem and palmate frond. The fronds of the former may be seen floating on the water at very low spring tides. The stems of the latter are well known as "Tangle," often six to eight feet in length, and as thick as a man's wrist. The frond is known as bardarrig or red top. The tangle is torn up and

thrown ashore by violent gales in the winter. The bardarrig generally comes ashore in May and September, and from these two plants, drift kelp, the only valuable variety, is made. These five algæ form the commoner species, and of these I have particularly to speak. There are some other well-known varieties to which I may briefly refer. The *Chondrus crispus*, or carragheen, or Irish moss, grows low down on the rocks, and can only be reached at very low spring tides. The plant, bleached by exposure, is employed in jellies and puddings. It contains an important gelatinising principle, known as carragheenin, of which I have found 63.7 per cent. If this plant could be obtained in quantity, it would be largely used as a size for fabrics. A much better gelatinising material is the *gelidium corneum*, which is remarkable as furnishing the import known as Japanese isinglass, a product imported from China since 1856. This substance, gelose, is of great gelatinising power, but the jelly formed does not readily melt in the mouth. The following proportions were found by experiment to be required in making equal quantities of jelly. The melting points are also appended—

	Part.	Melting point of Jelly.
Gelose .....	1	90° Fah.
Isinglass .....	8	70° Fah.
Gelatine .....	8	60° Fah.
Carragheenin .....	9	70° Fah.

These two algæ are the only British species which are gelatinous, and several articles, such as *thào*, fucyne, etc., appear to be obtained from these plants. An Australian alga, *Enchemia spinosa*, is also a gelatinous species. On sandy shores the *Laminaria saccharina*, or sugar wrack, is often found attached to single pebbles, and washed ashore with them. It derives its name from an efflorescence of mannite, which is often found on the fronds. In working on a considerable quantity of this plant I found 7.47 per cent. of mannite. It does not exist in the plant originally, but is the product of fermentation. On sandbanks, the *Zostera marina* or grass wrack is common, forming immense submarine fields. This material is largely used for packing goods and stuffing mattresses. It is not a true alga, but a marine plant with flowers. It attracted a good deal of attention some years ago, having been proposed as a substitute for cotton during the cotton famine. The plant, however, contains very little fibre, though it makes a good paper, and several patents have been taken out with this object. The algæ generally have no fibre, but all yield a cellulose which makes a tough transparent paper. The tissue of the plant consists of simple cells, which in many varieties contain a coloured pigment. This cellulose fabric amounts to 10 to 15 per cent. of the air dry plant. Another well-known plant is the *Sargassum bacciferum*, or Gulf weed, which forms enormous floating masses in the Atlantic. Some gigantic species are common in the Straits of Magellan and the Falkland Islands—the *Lessonia* and *D'Urvillea utilis*—specimens of which are to be seen in the Natural History Museum at South Kensington. The *Rhodomenia palmata*, or *Dulse*, and the *Alaria esculenta*, or *Murlins*, and some others, are used to a small extent as food. There is no doubt that we largely neglect the algæ, as we do the fungi, as sources of food in this country. Both are highly nitrogenous, and amongst the algæ there are no species known to be poisonous. They form a large article of consumption in China and Japan, some specimens closely resembling our own *Laminariæ*. In a paper read before the Chemical Society, in 1873, I pointed out that the charcoal obtained from the common algæ resembles that of animal rather than of vegetable origin. I append analyses of three typical charcoals. The resemblance of those from bone and seaweed will be at once seen.



The ash of the latter differs in containing more carbon and carbonate of calcium and magnesium and less of phosphates:—

	Wood.	Seaweed.	Bone.
Water .....	6.2	2.6	3.0
Carbon .....	92.0	63.0	9.2
Ash .....	1.8	34.4	87.8
Nitrogen .....	100.0	100.0	100.0
	0.20	0.92	0.25

The five common species first referred to are those used for manure, and in the making of kelp. The application of seaweed for manure is general on all farms adjacent to the sea, and a much higher practical value is given to it than the analysis of it would warrant. It usually contains at least 80 per cent. of moisture, so that four tons of water are carted to the land with every ton of manure, and even if dry, the saving of carriage is not great, as it is then very bulky. The value is partly mechanical, being rather to make than to manure the soil. The manufacture of soil and the cartage of water are both useful but expensive. Kelp waste, which contains all the phosphates of forty times its weight of seaweed, is quite unsaleable. Kelp for upwards of a hundred years has been the principal outcome of these algae and it has realised in bygone times large revenues to the highland proprietors, and has also been the chief support of thousands of poor crofters and cottars in Ireland and the West Highlands. Kelp was introduced about the middle of last century, and was employed in commerce for the soda it contained. At the beginning of the present century it was worth from £20 to £22 per ton, and the western isles of Scotland alone produced 20,000 tons. The importation of barilla then began, and during the twenty-two years ending 1822 the average price was only £10 10s. The duty was then taken off barilla, and kelp fell to £8 10s., and in 1823, on the removal of the salt duty, it fell to £3, and in 1831 to £2. From then till 1845 it was still used in the soap and glass factories of Glasgow. There was at one time a large chemical work in the island of Barra, built by General McNeill, for the manufacture of soap from kelp. Two tall octagonal chimneys were standing when I first visited the island, about twenty years ago. In 1845 the manufacture of iodine commenced in earnest, and kelp was again in demand. But the kelp required was not the same; that which contained the most soda contained the least iodine. Moreover, it became valuable as a source of potassium chloride—the richer kelps in iodine containing the most—and this salt was worth at one time about £25 per ton. The discovery of the Stassfurt mineral speedily reduced this price to about a third, and the further discovery of bromine in this mineral also reduced the price of that element from 38s. per pound to 1s. 3d. per pound, or about its present value. The amount of bromine produced from kelp is small, about a tenth of the iodine; and the total produce of France and Scotland is enormously exceeded by that of Germany alone, and a considerable quantity is also now produced in America. The manufacture of iodine from the mother liquors of the caliche of Peru has now assumed large proportions, and greatly reduced the price of that element, a substance which has ranged in value during the last forty years between 4s. and 40s. per pound. The present price is 5s., at which its production from ordinary kelp is unremunerative. The principal products of kelp and the quantities obtained per ton, over an average of 24,000 tons are as follows:—

	ewt.	%
Muriate (95 per cent. potassium chloride) .....	5	or 25
Sulphate (75 per cent. potassium sulphate) .....	1.8	or 9
Kelp salt (sodium chloride) containing sodium carbonate = 8 per cent. Na <sub>2</sub> O .....	3.8	or 19
	10.6	53

Iodine 12½lb.

When it is considered that it requires twenty tons of wet seaweed for every ton of kelp, it is astonishing how small a portion of the plant is utilised in this way. The kelp burner in the good old times only received £2 per ton for his labour; this is 2s. per ton for every ton of seaweed, which has to be cut, brought ashore, dried and burned, and even then all the labour may be lost by wet weather. Five tons of air-dry seaweed of the best quality, secured without rain, or 20 tons of wet weed, yield one ton of kelp. If made of the finest drift *Laminaria*, this may contain 15lb. of iodine to the ton of kelp (or '66 per cent., or '033 per cent. on the original weed); if made of fucus, it may be reduced to 3lb. (or '13 per cent., or '0066 per cent. on the original weed) or even lower. The proportion will vary between these limits according to the mixture of weeds burnt; but in all cases, from the great heat of the kelp fire, at least as much iodine is dissipated as is retained in the kelp. The great heat also reduces the sulphates, in the presence of carbonaceous matter, to sulphides, requiring considerable expenditure of oil of vitriol to decompose them, so that sulphur is one of the by-products in kelp lixiviation, and this is perhaps the only manufacture in which we reverse the ordinary process and make sulphur from sulphuric acid. Moreover, the fluidity of the molten mass admits of much adulteration. Large quantities of shingle and sand can be easily incorporated with it. So much has this been the case that the kelp ton used to contain 22½ cwt. to allow for adulteration. It is not uncommon to find a block of granite carefully varnished over with a thin veneer of this material imported as kelp. Our Irish friends are specially partial to lucrative practical jokes of this description. I am convinced nothing will be done to really utilise these seaweeds until we can make it worth while to import the raw material. There can be no adulteration then, and if an adequate price can be paid for it the gathering of seaweed would again become a very large industry. It has the advantage of employing the women and children of the rather numerous families in the Highlands and the West of Ireland, as all can easily work at it. There is no reason that any of the weed should be lost by rain, as it can all be preserved in silos. I have had quantities of the *Laminaria stenophylla* (the most difficult to preserve and the easiest destroyed by rain) preserved for several months in this way, perfectly fresh, and still containing 83.8 per cent. of water. The red weeds are mostly washed ashore by Atlantic storms, and the supply, although sometimes enormous, is not continuous. But the black weeds grow in great profusion in the sheltered lochs in the Hebrides and the West of Ireland, where they can be collected all the year round. Moreover, these do not suffer materially by exposure to rain. I have known 10,000 tons to be cut in one loch alone during a few weeks of summer. Four hundred thousand tons was in former years the annual collection in the Hebrides alone. This large amount of raw material is now quite unutilised. Kelp forms 5 per cent. of the plant, and as only half of this is soluble, the amount really utilised is 2½ per cent. By estimating the amount of iodine in the original weed and in the kelp obtained from it, the loss of iodine is easily estimated; but I prefer to illustrate this by a practical experiment conducted some time ago in North Urst. Eight tons of tangle were divided, four tons were burnt with the greatest care into kelp, and four tons were carbonised in an iron retort with the following results:—

	Cwt.	Percent.
Kelp produced .....	15	= 18.7
Charcoal .....	30	= 37.5
	Per cent.	Lb. of Iodine per ton.
Kelp contained salts .....	41.5	17.7
Charcoal contained salts .....	33	19.5

	Cwt.	Iodine, lb.	Per ton of Tangle.
Kelp produced salts ....	8'67	13'27	3'31
Charcoal produced salts	8'77	29'25	7'31
Loss in kelp burning ..	2'10	15'98	4'50

The loss cannot be less than this, and is generally a good deal more. Taking the *Laminaria digitata* and the *Laminaria stenophylla* as the plants most largely used for kelp making, these usually contain from 6lb. to 8lb. of iodine in the air-dry condition, if burnt into kelp, the usual yield from five tons is 18cwt., containing 9cwt. of salts and 15lb. of iodine. If carefully burnt into ash in brick ovens, with limited access of air, the yield is 20cwt., containing 11cwt. of salts, and 28lb. of iodine. If carbonised in perfectly closed retorts the yield is 36cwt. to 40cwt., containing 36lb. to 40lb. of iodine, 15cwt. to 16cwt. of salts, and about 30cwt. of residual charcoal. This is a great improvement, introduced by myself twenty years ago, but it still sacrifices the bulk of the plant. Moreover, it necessitates the erection of works in different parts of the shores, and involves long carriage of material.

I have found, however, that these salts and a great deal more can be easily extracted from the seaweed direct by simple maceration in cold water, and the process leaves the plant otherwise unaltered for further operation. Cold water extracts from the same quantity of material 33cwt. of soluble matter, containing 21cwt. of salts, and 30lb. to 40lb. of iodine, and 12cwt. of sugar, gum, dextrine, etc., leaving 77cwt. of material for further treatment. These salts are similar to those obtained from kelp, but contain no sulphides. They generally also contain calcium sulphate and magnesium chloride. I append analyses of these salts from two sources, and of the salts from the residual weeds after extraction by water.

	<i>Laminaria stenophylla.</i>	<i>Fucus vesiculosus.</i>
Potassium sulphate .....	9'72	23'62
Potassium chloride .....	31'79	13'71
Sodium chloride .....	48'67	58'20
Magnesium chloride .....	5'74	—
Calcium sulphate .....	1'93	4'33
Sodium hydrate .....	1'13	—
Sodium iodide .....	1'79	1'12
	99'95	99'98
Salts in residual dry weed ..	2'32	5'24
Potassium sulphate .....	35'27	29'41
Potassium chloride .....	6'72	—
Potassium carbonate .....	5'00	—
Sodium carbonate .....	49'97	4'5
Sodium sulphate .....	—	47'58
Sodium iodide .....	2'63	2'2
Calcium sulphate .....	—	9'34
Magnesium sulphate .....	—	11'76
Magnesium chloride .....	—	1'30
	99'49	100'09

It will be observed that the salts from these plants materially differ in composition, the first being typical of the *Laminaria*, the second of the *Fuci*. The following is an analysis of a lot of 2cwt. of salts fished from the water solution on evaporating down, a mother-liquor 54° Tw. being left :—

Water .....	22'70	
Volatile carbon, etc. ....	12'60	Dry
Salts .....	51'92	77'30
Fixed carbon .....	2'90	insoluble
Ash .....	9'88	12'78
	100'00	

#### ANALYSIS OF SALTS.

Potassium sulphate .....	14'20
Potassium chloride .....	27'81
Sodium chloride .....	55'11
Sodium iodide .....	1'69
Calcium sulphate .....	1'18
Magnesium chloride .....	nil
	99'99

#### ANALYSIS OF ASH.

Calcium and magnesium phosphates ....	52'40
Calcium carbonate .....	4'10
Magnesium carbonate .....	20'15
Magnesia .....	18'84
Sand, etc. ....	4'20
	100'00

It will be seen here that the magnesium chloride has been decomposed, the magnesia being found in the insoluble ash. After extraction of the salts the residual weed, which is unaltered in appearance, contains a peculiar principle to which I have given the name of Algin. This may be often seen in the natural state on the long flat fronds of the *Laminaria*, which, after exposure to rain, will be found to contain sacs of fluid. These are derived from the endosmosis of the water through the outside membranes dissolving this glutinous principle. Upon opening one of these sacs a neutral glairy colourless fluid escapes. It may often be seen partially evaporated on the surface of the frond as a colourless jelly, which adheres to the finger and may be drawn out in long threads. The vesicles of this fluid are best seen in the *Laminaria stenophylla*. The large tangles and the *Fuci* do not exhibit this appearance, although all contain the substance in large quantity. If the liquid be evaporated to dryness, it presents the appearance of scales like albumen, but is not then soluble in water, except on the addition of a little alkali. The natural fluid thus obtained is miscible with water, but coagulated by alcohol and mineral acids. It contains calcium, sodium, and magnesium in combination with a new acid, alginic acid. If the fronds be macerated in dilute hydrochloric acid, they become much swollen by the setting free in the interior of this acid in the form of a colourless insoluble jelly. To extract this substance the weed is treated with cold water containing sodium carbonate. In twenty-four hours the whole plant disintegrates and forms a thick gelatinous mass. The cellular fabric of the plant is completely broken up. This mass, containing about 2 per cent. of total solid matter, is too solid to pour out, and on account of its extraordinary thickness and the minute character of the cellulose it is difficult to deal with, and extremely difficult to filter. By gently heating it, and if necessary diluting it, it can be filtered through a coarse linen sacking in the form of a Taylor's filter. Pressure is not at this stage practicable. The cellulose is left behind in a gelatinous mass, amounting when dry to 10 to 15 per cent. of the air-dry plant. The solution contains, in addition to the algin, in the form of alginate of soda, some mucilage and dextrine. It is precipitated by sulphuric or hydrochloric acid, and the alginic acid, which separates in albuminous flocks, is easily washed and pressed in an ordinary wooden screw press, or in a filter press. In this condition it forms a compact cake not unlike new cheese, which can be stored in a dry room for any length of time. If not sufficiently white a little bleach solution is added during the precipitation, which makes it perfectly colourless. When required for use it is redissolved to saturation in solution of carbonate of soda in the cold, or with a gentle heat, carbonic acid is disengaged, and alginate of soda again formed. Carbonates of potassium and ammonium, or the corresponding bicarbonates, may also be used. The caustic alkalis do not answer so well. The solution is so thick that it cannot be prepared above 5 per cent., and then it will not pour. It may be alkaline, neutral, or acid, and it keeps very well if properly prepared. The solution first obtained from the plant requires precipitation within a few days. Some fermentation often takes place, which converts the whole of the algin apparently into a dextrinous substance. I do not yet know the cause



of this change, as some of the solutions have kept good for twelve months. The evaporation of this solution requires a similar treatment to gelatine or glucose, and must be effected on thin trays or slate slabs in a drying-room with a current of air, or by a revolving drum dipping in the solution and heated internally by steam. The algin so made resembles gelatine in appearance, but is much more flexible. It is perfectly soluble in cold water. The solution, if alkaline, can be rendered neutral by hydrochloric acid, but an excess at once precipitates it. A two per cent. solution becomes semi-solid heated in this way. It is precipitated or coagulated by alcohol, ethylic and methylic, and acetone; by the following acids—hydrochloric, nitric, sulphuric, sulphurous, phosphoric, citric, tartaric, lactic, oxalic, picric, and sulpho-indigotic; by salts of barium, calcium, strontium, aluminum, chromium, uranium, antimony, zinc, cadmium, cobalt, nickel, copper, bismuth, tin, silver, and platinum; by iron sulphate (white), iron perchloride (brown), mercury protonitrate and pernitrate, lead acetate and subacetate; by lime water and baryta water. It is not coagulated nor precipitated by ether, glycerine, amyl alcohol, starch, or sugar; nor by the following acids—acetic, boracic, tannic, formic, carbolic, benzoic, butyric, gallic, pyrogallic, succinic, arsenious; nor by salts of magnesium and manganese and the alkalies; nor by sodium and potassium silicate; nor by potassium chromate and bichromate, ferro and ferricyanide, permanganate; nor by sodium stannate, succinate, biborate, and tungstate; nor by bromine, iodine, or chlorine; nor peroxide of hydrogen; nor by lithium carbonate, ammonium molybdate, or tartar emetic. It is remarkable that it should precipitate the other alkaline earths, but not magnesium, and that it should not precipitate mercury bichloride, and that it gives no precipitate with the alkaline silicates. It is distinguished from albumen by not coagulating on heating; from gelose by not gelatinising on cooling, and by containing nitrogen, and by dissolving in cold weak alkaline solutions; from gelatine by giving no reaction with tannin; from dextrine, gum arabic, and pectin (which it most resembles), by insolubility in alcohol and dilute acids, and by being nitrogenous. Alginic acid dries up to a hard horny substance: some varieties of it are very like horn. It is almost impossible to obtain it perfectly pure. A well-washed specimen contained 3.68 per cent. of ash. The following is an analysis of the ash:—

Potassium sulphate.....	7.33	Soluble, 54.13
Sodium sulphate.....	9.60	
Sodium chloride.....	29.88	
Sodium carbonate.....	7.31	
Sodium iodide.....	.17	Insoluble, 45.87
Calcium sulphate.....	.17	
Calcium carbonate.....	35.96	
Iron, alumina, phosphates...	6.26	
Silica, etc.....	3.18	
	100.00	

The following is the analysis of algin or sodium alginate:

Water.....	17.13—17.13		
Alginic acid.....	67.58	Ash, 22.9 per cent. containing sodium carbonate 18.32	
Soda.....	10.71		
Alkaline salts.....	2.98		
Calcium carbonate.....	1.00		
Alum, alumina, etc....	.60		
	100.00	100.00	
Gum Arabic contains		Gum Tragacanth contains	
Water.....	17.6	Water.....	11.0
Arabin.....	70.1	Arabin.....	53.3
Alkaline and earthy salts.....	12.0	Bassoin.....	33.3
		Alkaline and earthy salts.....	2.1
	100.0		100.0

A carefully-dialysed specimen of algin, containing 19.69 of ash, was submitted to combustion by Prof. Ferguson, and gave the following numbers: Carbon

44.39, hydrogen 5.47, nitrogen 3.77, oxygen 46.37. The numbers approach nearly to those of chitin, but I cannot consider the substance as sufficiently pure to make these data more than approximate. The viscosity of algin is remarkable. It was compared with gum arabic and with well boiled wheat starch. The gum was used in a 25 per cent. solution (the British Pharmacopœia mucilage is over 50 per cent.); the starch, as in the British Pharmacopœia, was 2.5 per cent. The algin was tried first at the same strength, but it would not run through the viscometer tube at all, so the strength was reduced to 1.25 per cent. The rates of flow were as follows for the same quantity, 550grs. measure:

Gum arabic solution	%	took	sec.	sec.
Wheat starch soln.	2.5	took	20 = 1 in	8 or as 2.66
Algin solution.....	1.25	took	130 = 1 in	112, or as 37.33

So that the algin has about 14 times the viscosity of starch and 37 times the viscosity of gum arabic. Alginic acid is slightly soluble in cold water, more so in boiling. Cold water dissolves 0.248 per cent.; boiling water, 6.24 per cent. Sodium alginate heated for some time in solution, if the soda be in excess, is partly changed into a body resembling dextrine. The proportion of sodium carbonate used is one-tenth of the weight of the air-dry weed, and I append table of quantities of dry alginic acid obtained from different air-dry weeds. The yield of ordinary algin on this dry acid is about 150 per cent. The weight of the cellulose is also given:—

	Laminaria Digitata. Stem.	Laminaria Digitata. Frond.	Laminaria Stenophylla. Stem.	Laminaria Stenophylla. Frond.
Water.....	37.04	44.0	34.50	40.02
Algin.....	21.00	17.85	25.70	24.66
Cellulose....	28.20	11.00	11.27	15.06

I append also analyses of the dry cellulose.

	Laminaria Digitata.	Laminaria Stenophylla.	Gross Vesicular.
Yield of charcoal contain- ing.....	38.36	..	44.62
Salts.....	11.08	..	11.06
Carbon.....	12.72	..	15.93
Ash.....	11.55	..	17.63
	38.36	36.41	44.62
Iodine.....	.12	.06	.05

I have thus accounted for the greater part of the total constituents of the plant, but not for all. The three principal components are the salts, the algin, and the cellulose; but the water solution also contains mucilage and sugar, and the sodium carbonate solution contains a modified dextrine. These substances are still under investigation. It is obvious that we are not at all confined to the method of working here described. There are several other methods which may be adopted, one of which may ultimately prove more economical. The weed may be treated direct with the sodium carbonate, and after precipitation, either by sulphuric or hydrochloric acid, the sulphate or chloride of sodium may be recovered with the other salts, after neutralising the excess of acid with lime or limestone. Or calcium chloride, a waste product, may be employed as the precipitant, or a salt of alumina may be used. With either of these agents the precipitate falls to the bottom of the liquid, whereas with the acids it rises to the surface. The calcium alginate forms a very compact and easily pressed cake, which can be decomposed by sodium carbonate. The aluminium alginate is soluble in caustic soda, and this compound is therefore easily made and dissolved. It forms a neutral compound similar to algin, but harder and stiffer. This substance is still under examination, as its constitution and properties suggest some important applications. Algin forms also a singular compound with shellac, not unlike

guttapercha. Prepared from its solution in ammonia it is obtained in thin sheets, possessing great toughness and flexibility. By passing through an acid bath it can be rendered quite insoluble in water. Algin has the same effect as gelatine or bichrome, the film becoming insoluble after exposure to light. The silver alginate also darkens very rapidly on exposure to light. These properties suggest its use in photography, especially in photographic engraving.

*Commercial Applications of Algin.*—The properties of algin in the soluble form are those of a very viscous gum, drying to a transparent elastic film. As a size it goes much further and does more work than starch or any of the ordinary gums. In sizing fabrics, compared with starch it is tougher, more elastic and transparent, and though less rigid, fills the cloth better. It mixes in all proportions with starch, dextrine, and other gums, and may be used in combination with any or all of these. Unbleached it forms a cheap dressing for winseys and dark goods. When passed through an acid bath the coating becomes glossy, and insoluble in water. Lime water, salts of calcium or aluminium, and many metallic salts may be employed instead of acid for this purpose. One of its important properties is the ease with which it is rendered insoluble. It is perhaps the only efficient vegetable mordant yet discovered, as it precipitates alumina, iron, and other metals completely from solution, the compounds having great attraction for the ordinary colour used in dyeing. The soluble alginate of alumina will, I believe, prove a valuable dressing material for many purposes, as it gives great stiffness to fabrics prepared with it. Algin is a valuable food material, and in its pure state has a pleasant marine flavour. It will make an excellent thickening for soups. For jellies it requires the addition of a little gelatine or isinglass. I suggest it particularly for the making of jujubes. Many singular compounds may be obtained by mixing a solution of algin with another solution, for instance sodium silicate, and adding the mixture to another solution, for instance, calcium chloride, which forms an insoluble compound with both. In this way insoluble vegetable matter is introduced into calcium silicate. In the same manner phosphate and alginate of calcium can be made in various proportions, forming a compound similar to bone. The agglutinating power of algin is considerable, and enables us to convert into solid blocks many substances which cohere with difficulty, for instance silica, ordinary sand, limestone, magnesia, phosphate of lime, alumina, steatite, plumbago, charcoal, and many other non-adherent substances. Its application to charcoal has already assumed considerable importance. Charcoal has been long known as our best solid non-conductor of heat, and no doubt it would have been employed long ago as a covering for steam boilers if it could have been made to cohere, in other words to stick, on the plates. The new boiler covering, carbon cement, contains 97 per cent. of charcoal and 3 per cent. of algin, and as the best charcoal for the purpose is made from seaweed, it is remarkable that the whole covering is made of that material. It forms a light, efficient, inodorous, and cheap material for covering steam boilers and piping, and has already been largely used. As to its non-conducting power, the engineer of one of our large Atlantic steamers told me he could lay his face against it. The solution of algin has a wonderful effect in arresting incrustation in steam boilers. We are not troubled with this complaint in Glasgow, where Loch Katrine water is used; but in other places, where the waters are hard, many such fluids are employed, and indeed are absolutely necessary if fuel is to be economised. Most of these fluids are organic compounds combined with alkalis. I am

indebted to my friend Mr. Spiller for suggesting the use of algin for this purpose, he having been the first to introduce a good boiler fluid some years ago. He has found this one highly efficient in precipitating the lime in such a fine state of division that it can easily be blown off from the boiler. Algin can be used for fining spirits and emulsifying oils. The compound with shellac may be useful for electrical purposes. The insoluble form of algin in the dry state resembles horn, the calcium alginate resembles bone, both are capable of polish, and are easily turned in the lathe. The cellulose also, although in the wet state available for paper, when dry is extremely hard, and can be made very dense by pressure. It also turns and polishes well, and may be useful with or without the addition of algin for some of the purposes for which horn and bone are used. Since my first paper on the subject was published, I have been much surprised by the number of letters that have reached me from different parts of the country, all speaking of the great want of some such substitute. A similar heavy correspondence has also convinced me that a good sizing and dressing material for fabrics is a great desideratum. Many appear dissatisfied with the present supply, and have complaints to make about each and all of them. Something new is evidently wanted, and there is apparently room for a great deal of it. Such a want will, I hope, be supplied by this new product. At any rate, in developing it we are endeavouring to open up a new field, which still requires, and will, I believe, repay, further exploration.

#### DISCUSSION.

Dr. WALLACE said: Mr. Stanford has taken up the chemistry of algin both scientifically and practically, and for a good reason, because he is both a scientific and a practical chemist. The substance will remind those scientific chemists who are present very much of pectin, for although it is an entirely different thing from pectin, there is a resemblance which forces itself upon us. What pectin is in ordinary vegetables and fruits, algin is in marine plants, and when it is considered that pectin has been the study of some very distinguished chemists such as Braconnot, Regnault, Frémy, and a great many others, and that it is still the subject of investigation, although numerous products have already been obtained from it, we cannot wonder that our friend has not exhausted the chemistry of this very interesting and important substance. Mr. Stanford has not given us the ultimate composition of algin, probably he has not yet been able to obtain it in a perfectly pure condition, and as we all know, analyses of imperfectly purified proximate principles only lead to difficulties. I fancy, Mr. Stanford, such is the case, and that you have not procured it in a fit state for chemical analysis. It appears to be evident that this algin is capable of many changes, and I have no doubt forms acids and compound acids in the same way that pectin does. It seems also to have the property of combining with numerous bases, but perhaps Mr. Stanford has not been able to ascertain whether the precipitates thrown down are definite alginates of the various bases, or in some cases are simple precipitates of algin itself. I have no doubt it would take a very long time to exhaust this subject, and probably it may require other workers besides Mr. Stanford to complete the investigation. There is one question I would like to ask Mr. Stanford, and that is whether the alginate of alumina is soluble in ammonia. If this compound is soluble in ammonia, the solution might be employed for many useful purposes, for instance, as a kind of varnish in cases where the alginate of soda or potash would not be applicable.



Mr. CHRISTIE: I think we are all very much indebted to Mr. Stanford for the time that he has devoted to this interesting subject, and for the valuable paper that we have just listened to. I may say that I have made some experiments to determine how far it could be used as a substitute for starch in the finishing of cotton fabrics. Alginic acid in the free state is capable of holding a large quantity of water, and in that condition has a fibrous appearance not unlike a transparent paper pulp. It is quite insoluble in water, and from its fibrous structure cannot in the free state be distributed like starch in a thin film in the finishing of cotton prints, etc.; therefore we must resort to the soluble form, and that in the shape of a salt. The soda salt can be applied to cotton goods with good results, but the properties are entirely different from those given by starch. It fills the goods well but has no stiffening property, or at all events but very little. The finish is more of the character of a soap filling, imparting to the goods a thick, clothly, and elastic feel without the stiffness imparted by starch. There is another application of the alginate of soda that occurred to me might be of some interest, namely, in the fixing of mordants such as alumina or iron upon cotton fibre. I find, so far as I have gone with the experiments, very encouraging results. I believe a very large application will be found for the alginate of soda as a dunging substitute. The mordants when precipitated seem to have full dyeing power, the results indicating that this substance is capable of taking the place of cowdung as used in print and dye works; also as a dunging substitute it will rank with arseniates, phosphates, silicate of soda, and a number of other salts which are now largely used for the precipitation of mordants previous to the dyeing of cotton fabrics and yarns.

Mr. Tervet: I should be pleased if Mr. Stanford would inform me how far this algin could be made applicable as a substitute for glue. It is well known that in casking paraffin oil, all the staves have to be glued inside to render the wood impervious to oil; and I would like to know if it could be made applicable for that purpose.

Mr. Pattison: It may interest members to know of some experiments which have been made with algin. It was hoped that it might be used for stiffening lace, instead of starch, and it has been tried by one of the leading firms in Nottingham for that purpose. In its present state, however, it gives a finish which is considered too soft. A calico printer here has printed some pigment colours with it, and then passed the cloth through an acid bath, but the results have not been satisfactory, probably owing to the fact of the precipitated algin being easily soluble again, in even weak alkaline solutions. Algin has been tried as a glaze for paper instead of gelatine, and also as a substitute for glue in paper box covering. It seems to answer for the latter purpose, but as no market price has yet been fixed, its relative value as compared with glue has not been estimated. A good deal of animal size is used by calico printers, and it is probable that algin, if cheap enough, might be used with advantage in the dye-bath, instead of that substance.

Mr. Tatlock: I should like to ask Mr. Stanford whether algin contains sufficient nitrogen, and whether, in his opinion, it could be produced cheaply enough, for the manufacture of prussiates? It seems to me that if carbonate of potash was the alkali employed for its extraction that would not be lost, as it is one of the essential ingredients used in the manufacture of these products. Mr. Stanford has expressed the hope to see kelp burning resuscitated on the shores of Scotland and on the shores of the north of Ireland, but it seems to me, in the present

condition of the potash trade, and of the nitrate of soda trade of South America, that we are very far from seeing that. There is practically an inexhaustible supply of potash salts in Germany, and of iodine in the crude nitrate of soda of Peru, and with these two together we can buy at present, I might say, every product except algin—if Mr. Stanford will allow me—which is a new substance, at a cheaper rate than it can be had from kelp. It seems to me, with regard to the nitrate of soda trade, there are only two lines for that business to follow. There must either be a "combination," and the output restricted to a large extent, so that the nitrate will be confined to such uses as the manufacture of nitric acid, and for vitriol making—and for these purposes it would command any reasonable price. But Mr. Stanford knows the difficulty of making and of continuing "combinations," and, failing this, the temptation will be for the nitrate of soda manufacturer to turn out as much nitrate as possible with the object of competing with the large amount of sulphate of ammonia in prospect. When that largely increased production appears, we should have a strong competition between it and nitrate of soda; and then, of course, if we have an increased production of nitrate of soda, we should expect also an increased production of iodine, making it extremely improbable, to my mind, that we shall see the kelp-burning industry revived to any large extent upon our shores.

#### REPLY.

Mr. Stanford, in reply to the previous speakers, said: Dr. Wallace has mentioned a substance which algin appears to approach in some of its reactions, but about this time last year I published a centesimal composition of it—with the nitrogen it contained—and shewed that the only substance at all near it was chitin the main constituent of the carapace of the crustacea, of which there is a specimen on the table prepared from the crayfish. But it differs entirely from chitin, for that is insoluble in potash, whereas this is quite soluble. Dr. Wallace has made a very pertinent remark about the extreme difficulty of getting this substance perfectly pure, and the only reason I have not published another analysis is that I do not consider I have got it yet in a perfectly pure state, and I may never be able to so obtain it. Gum arabic has only very recently been really worked out. It has been worked on for many years, but every chemist who has touched it, up to within the last few months, has left it almost as he found it, simple as the substance appears to be. The fact is, these vegetable principles differ materially, according to the age of the plant, the time of the year, and many other conditions, all of which would affect a substance of this sort, and therefore it may be extremely difficult—it may even be impossible—to obtain it in a state of absolute purity. I am not quite sure whether alginate of alumina is soluble in ammonia. I should think not, but it is a question for experiment, and I shall soon ascertain. As to the glueing of casks—I think that was another question—I do not think this substance would be equal to glue in adhesiveness. It is much more elastic, and I should think not so good as glue would be for ordinary glueing purposes, although it might be quite suitable for lining casks. With regard to the prussiate question, that Mr. Tatlock has spoken of, I can only say that I do not want to reduce it to a price that will enable me to compete with prussiate manufacturers. And with regard to his remarks as to the kelp industry, he must have taken me up wrong. I have stated distinctly that if we have to deal with kelp for the manufacture of iodine and potash salts, we shall infallibly be beaten by other manufacturers—or "diggers" as I may call them—who are able to dig it from

the earth. If they can dig iodine, as they do practically from the earth, then we cannot compete with them; but I have yet to see the country which has succeeded in digging anything like that new product algin from the earth, and when they do so I shall be quite ready to admit that they have beaten me. At present that is not the case.

MR. ED. C. C. STANFORD IN THE CHAIR.

## ON ALKALI-PROOF VESSELS.

BY PROF. DITTMAR F.R.S.

PREPARATIVE or analytical operations with caustic alkalis are beset with characteristic difficulties, which, although in a given case they may be of a purely financial nature, are none the less keenly felt. In the course of my laboratory practice I have made repeated attempts to turn these difficulties, and as one result, I have come, since about two years, to use basins of malleable nickel for operations with aqueous caustic alkalis. Nickel, until lately, was known in commerce only in the form of those "nickel-cubes" which, although they might contain as much as 99.5 per cent. of the pure metal, were always utterly devoid of plasticity: they broke under the hammer. That this brittleness is not an inherent property of the metal had long been proved by A. Richter, who, in an early decade of this century had obtained the metal in a form in which it was highly malleable and ductile. Yet malleable nickel remained unknown to the arts until Fleitmann, in 1879, made a remarkable discovery. Finding that even the purest nickel which he was able to produce in his works yielded unworkable ingots, he suspected that the brittleness of the metal was owing to the presence in it of occluded carbonic oxide. He accordingly tried the effect of fusing it up with a small proportion of magnesium, and found that as little as one-eighth per cent. of this addition cured the defect, and imparted to the regulus such a high degree of plasticity that it could be rolled into the thinnest sheet and drawn into very fine wire. He also found that nickel at a red heat can be welded together with iron or steel, and that a compound plate thus produced stands rolling out to any extent without breach of continuity. Since this discovery Mr. Fleitmann's firm have made a business of the production of kitchen utensils, which are nickel inside and outside, with a core of iron between the two. These utensils, as you see by the specimen I exhibit, are almost as pretty as silver ones, and practically as little given to spontaneous oxidation as these. Over copper vessels they offer the advantages of being harder and absolutely innocuous. When I read Dr. Fleitmann's publication it at once struck me that basins made of malleable nickel would be the very thing for operations with caustic alkali leys, and I caused a Glasgow silversmith to procure a supply of Fleitmann's metal and try to make it into a basin for me. My friend was not successful; the metal, although malleable at first, became stiff and intractable under the hammer. A few years later, when the idea again turned up in my mind, I applied to Messrs. Johnson, Matthey, and Company, of London, and they, after one or two failures, succeeded in producing this little basin (exhibited), which since has been used by my students hundreds of times, chiefly for the separation of iron and alumina. The basin, as I hardly need say, was not always very tenderly handled, and yet, as you see, it has quite retained its original shape, and almost its original polish. This large basin (pointing to another exhibit),

which boils a litre of liquid, was procured from the same firm about eight months ago. It looks as if it came straight out of the workshop, although it has been used many a time for preparative purposes. The metal of these basins, in regard to its power of resisting the action of caustic-alkali ley, is quite at a par with platinum; and it is certainly more convenient (apart from its being far cheaper) than silver; it has a greater permanence of form, is quite devoid of porosity (which fine silver is not), and does not conduct heat so inconveniently well. A nickel basin half-full of boiling caustic potash ley can be handled with the naked fingers with impunity. In the present connection the only inconvenience of these nickel vessels is that, for instance, a patch of oxide of iron which may stick to the basin must be removed mechanically, because the metal is attacked by aqueous acids, although it is dissolved far more slowly than iron is. It must also be stated that nickel basins do not stand ammonia, in the presence of air at least. That nickel basins would be proof against fused caustic alkali I never expected, but I was curious to ascertain what strength a caustic alkali ley must have attained before it begins to attack the metal. A solution of commercial caustic potash, containing 44 per cent. of real KHO was kept in a nickel basin over a hot water-bath for over two hours, and then allowed to stand in the basin cold over night. From the loss of weight which the solution had suffered by evaporation, the finally present percentage of pure KHO was calculated to be=59.6. Yet the basin had remained bright, and lost only 2 milligrammes of its weight. A second experiment was made with a stronger solution of caustic potash, which was kept in gentle ebullition by means of a gas lamp, the water lost by evaporation being replaced from time to time, judging by the eye. Time of exposure, this time, about one hour; original strength=58.8 per cent. of pure KHO; final strength (contrary to intention) greater, namely, =68.6 per cent. The basin had lost 10 milligrammes, from an exposed area of about 90 square centimetres, and the ley contained a trace of dissolved nickel. The limit of safety seems to lie somewhere near 60 per cent. of real KHO. This second experiment was repeated with a caustic soda (instead of a potash) ley. The concentration rose from 58.5 per cent. to (finally) 61.5 per cent. of real NaHO. The basin was slightly but perceptibly attacked; it had lost 4.5 milligrammes in weight. To pass now from the aqueous to the dry reagents. Caustic alkali, when in a state of fiery fusion, as we all know, attacks even platinum very badly. Of ordinary metals, in fact, only gold is quite proof against its powerfully corrosive action.\* But gold is very expensive, and, what is worse, very soft. Chemists therefore, generally try to get on as well as they can with fine silver, although this metal, as we know, is not quite proof against the fused reagent, and besides is inconveniently soft and porous. It sucks in fused caustic alkali like a sponge. Even a small silver crucible, after having been used for a caustic alkali fusion, and carefully cleaned, always weighs quite a number of milligrammes more than it did originally. I once had a silver crucible which, although perfectly water-tight, allowed fused caustic potash to slowly filter through its walls. Some six years ago I tried to remedy these defects by alloying the metal with a little nickel; but I found (as Lampadius had done 50 years before) that silver refuses to amalgamate with nickel. But learning from the same authority that nickel readily unites with gold, I made a gold-nickel alloy, and added an

\* To make sure of the correctness of this general assumption, I fused caustic potash in a tray made of pure gold foil, and kept up a bright red heat for a considerable time. The gold remained perfectly bright.



excess of it to a relatively large mass of molten silver. There resulted an alloy which had at any rate the expected degree of hardness and elasticity, and Messrs. Johnson, Matthey and Company had no difficulty in bringing it into the form of a boat for me. This boat has served during the last six years for numerous determinations of organic nitrogen in water residues according to that method which Mr. Robinson and myself published conjointly some years ago, and has been found to hold out far better than, presumably, a plain silver one would have done. This means that it was not attacked by many fusions in it of a mixture of caustic baryta and caustic soda, carried out, however, in an atmosphere of hydrogen. The boat would be in use this day if I had not cut it up some time ago to ascertain its quantitative composition, which was found to be as follows—

Silver .....	91 per cent.
Gold .....	7 per cent.
Nickel .....	2 per cent.

100

With this receipt in their hands, Messrs. Johnson Matthey and Company made for me a new boat, and a crucible of the alloy. (Exhibited.) My main object in ordering the new articles was to compare the alloy in regard to resistance to fused alkali with pure silver; but when I was once at it I thought I might as well extend my inquiry to nickel and platinum, and did so. At first sight it appears rather singular that all ordinary metals are attacked by fused caustic alkali, although only a few—namely, tin, zinc, and aluminium—have the power of decomposing its water with evolution of hydrogen and formation of an alkaline metallate. This very forcibly suggests that, in the case of other metals than these three, the corrosive effect is due not to the alkali in itself but to the peroxide formed from it by the action of the atmospheric oxygen. When we fuse an ounce of caustic soda in an iron crucible it gets hopelessly contaminated with oxide of iron. The manufacturer, on the other hand, fuses his 10 tons in an iron pot, and produces a snow-white preparation, because in his case the proportion of peroxide produced, as far as it comes into contact with the iron, is vanishingly small. These considerations guided me in my experiments, which, as a rule, consisted in this, that the respective metal was used in the form of a boat, which, after having been charged with the reagent, was placed in a combustion-tube and heated to redness, sometimes in a current of air, sometimes in a current of hydrogen. In the case of the air experiments, a crucible was used occasionally instead of a boat, and, in the special case of platinum, part of the hydrogen trials even were carried out within a crucible provided with an inlet and an outlet tube, soldered, autogenically into the well-fitting lid. (Crucible exhibited.) The most important result of my experiments is that the metals examined—namely, silver, the silver-nickel-gold alloy, platinum, and nickel—at even a red heat, are all proof against fused caustic potash, soda, and baryta ( $\text{BaO}_2\text{H}_2$ ) in the absence of air. Hence if we want to disintegrate, say, a silicate by fusion with caustic potash or caustic baryta, we may use a platinum crucible, and consequently apply a bright red heat, if we only take care to maintain in the crucible an atmosphere of hydrogen. By means of the crucible which I exhibit I have had no difficulty in decomposing felspar with caustic baryta, so that the “fuse” could have been analysed for potash and soda as well as for the rest of the components. I have not made any such direct trials with caustic potash or soda as a reagent, but am in a position to state that the alkalis, when kept red hot in an atmosphere of hydrogen, fuse quietly, while in the

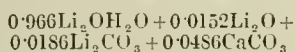
presence of air they boil up and are apt to run over the edge of the vessel. In cases where an atmosphere of hydrogen would be objectionable, as for instance in the case of tin-stone, nitrogen might be substituted for the more volatile gas, and, I have no doubt, would work as well or better. A crucible intended for such alkali fusions, however, should have a shorter outlet pipe than the one which I exhibit, and which, as I may remark in passing, was originally intended for work of an entirely different order. By the conjoint action, at a red heat, of fused alkali and air, all my metals were attacked, silver not excepted, although it must be admitted that it held out best, and appreciably better than even my triple alloy. But whichever of the metals was used, caustic potash always acted more powerfully than caustic soda. Caustic baryta ( $\text{BaO}_2\text{H}_2$ ), as far as my experience goes, is a stronger corroder certainly than caustic soda, and not necessarily milder in its action than caustic potash. On silver more especially it seems to act rather more energetically than potash does. To illustrate these general statements I will quote a few examples. The alloy crucible was charged with 19 grms. of pure caustic potash, and the reagent fused and kept at a red heat for some twenty minutes. After the surplus water had boiled off the liquid became black, probably through the decomposition of organic matter contained in the reagent,\* because the colour disappeared after a time. But the black colour subsequently returned, and became more and more intense through the action of the peroxide formed on the metal. The fuse, when dissolved in water, gave off a gas (probably oxygen) and yielded an abundant black precipitate containing (by analysis) 47 milligrammes of silver (Ag.) and 3.4 milligrammes of nickel. The crucible had lost 57.5 milligrammes of its weight. A fine silver basin when subjected similarly to the action of 26 grms. of the same potash (at a somewhat lower temperature) behaved similarly to the alloy-crucible; only, apparently, it did not yield so readily to the reagent. Yet the fuse when treated with water gave off gas and yielded a residue weighing 69 milligrammes, and containing, by analysis, 58 milligrammes of metallic silver. The basin lost only 6 milligrammes of its weight, showing that it had sucked in caustic alkali. The alloy crucible when treated with 9 grms. of caustic soda at a red heat for 30 minutes, lost only  $1\frac{1}{2}$  milligrammes of its weight. The fuse dissolved in water with only a slight gas evolution, and formation of a small black precipitate. The silver basin, under similar circumstances, behaved pretty much the same way, but it gained 7.5 milligrammes in weight, proving absorption of soda by its porous metal. A small fine silver basin (of about 35 millimètres diameter and 17 millimètres deep), when subjected to the action of caustic baryta, kept fused in it at a red heat, was strongly attacked, and yet, after having been cleaned, weighed 32 milligrammes more than before. In connection with the present subject one cannot help remembering the conflicting statements of different authorities in regard to the action on platinum of fused lithia. Since the time of Arfvedson, not merely lithia proper but even lithium compounds generally had been credited with quite a characteristically strong corrosive action on platinum at a red heat, until Troost made it out that lithia does not attack platinum unless it be contaminated with rubidia or cæsia, as it very often is in practice. As the proposition bears the stamp of such a distinguished name I felt inclined to accept it as correct, the more so as a few incidental laboratory experiences of my own rather tended to confirm it; but I did not feel quite sure, and accordingly inquired into the matter experimentally. To procure a lithia

\* It was “potasse à l'alcool.”

sure to be free from potash, rubidia, and caesia, I dissolved a supply of *Lithium carbonicum purum* from Trommsdorff in Erfurt, in hydrochloric acid, and after addition of an ample excess\* of chloride of platinum evaporated to dryness on a water-bath. The residue was taken up with a mixture of equal volumes of absolute alcohol and absolute ether, the liquor filtered off from the chloroplatinate produced, freed from its ether and part of the alcohol by distillation, and then evaporated to dryness. The residue was reduced in hydrogen at about 300° C., and then dissolved in ether-alcohol to remove the platinum and incidentally what there might be of chloride of sodium. This led to a chloride of lithium, which, although not sure to be quite free from sodium, could not have contained any of the more basulous alkali metals. To convert it into carbonate, without the use of fixed alkali, I evaporated it with a solution of the calculated weight of alkali-free oxalic acid to produce the acid oxalate, which was found to crystallise very readily. Rejecting the mother-liquor, I decomposed the crystals at the lowest possible temperature in a platinum basin, which, to my surprise, led to the formation of a large proportion of charcoal,† to which circumstance it may be owing that the platinum remained unattacked. The residual mixture of carbonate and charcoal was ground up with water, and carbonic acid passed into the mixture to produce a solution of bicarbonate, which was filtered and decomposed by evaporation on a water-bath, so that the bulk of the dissolved carbonate was obtained in the shape of a crystalline crust. This crust was collected, rinsed with water, and accepted as pure carbonate of lithia  $\text{Li}_2\text{CO}_3$ . Part of this carbonate was converted into lithia by the ordinary process of causticising with lime, the process being conducted in the large nickel basin, by means of pure lime made by igniting pure precipitated carbonate in a platinum crucible. The decanted lithia ley was evaporated down in a large platinum basin, when a deal of carbonate of lime or  $(\text{Ca}(\text{OH})_2?)$  separated out, which was allowed to settle and eliminated by renewed decantation. The thus purified liquor was then evaporated further in a silver basin, the dry residue fused, and preserved as lithia. It turned out a poor apology for real  $\text{LiOH}$ , as shewn by the following analysis:—

	Per cent.
Lithia ( $\text{Li}_2\text{O}$ ) .....	56.53
Carbonic acid ( $\text{CO}_2$ ) .....	5.57
Lime ( $\text{CaO}$ ) .....	5.13
Water (by difference) .....	32.77
	100.00

the composition of a mixture of the formula—



Both preparations acted strongly on platinum vessels when fused in them in the presence of air, and there did not seem to be much difference between the two in regard to the violence of their action. I content myself with quoting an experiment made with the carbonate in a platinum crucible of about 15 c.c. capacity. A quantity of the carbonate was placed in the crucible, and at first heated gently for a time to drive off any water or excess of carbonic acid. The residual dry carbonate ( $\text{Li}_2\text{CO}_3?$ ) amounted to 4.413 grms. The heat then was raised to and maintained at full redness for about an hour and a half, the lid being kept on. The fused carbonate crept up at the sides of the crucible, so that some, by

travelling along the lid, reached the frame and coloured it crimson. The crucible, especially at those parts of the side which were only, so to say, varnished over with the reagent, was very perceptibly attacked. To increase the effect, a coil of platinum foil was now placed in the crucible, the lid put "half on," and the heating resumed and continued for other two hours. The crucible and contents were now allowed to cool, and weighed, when it was found that the contents weighed 0.161 gm. less than the original carbonate. All the visible inside of the crucible had assumed a dark olive-green colour. To analyse the product the crucible with its contents was placed in a cylinder rigged up as part of an apparatus for the direct determination of carbonic acid. In it the product was decomposed by dilute sulphuric acid, the carbonic acid collected in a potash bulb and soda-lime tube and weighed. An aliquot part of the solution left served for the determination of the lithia as sulphate.

## RESULTS.

	Grms.
Original carbonate .....	4.413
Total residue in crucible at the end of the heating .....	4.252
Containing $\text{CO}_2$ .....	2.313
And lithia equal to $\text{Li}_2\text{SO}_4$ .....	6.543

This agrees with the assumption that the residue was a mixture containing

	Parts.
Carbonate of lithia ( $\text{Li}_2\text{CO}_3$ ) .....	91.49
Binoxide of lithium ( $\text{Li}_2\text{O}_2$ ) .....	7.49
Extra oxygen (by difference) .....	1.02
	100.00

The crucible and coil, after the sulphuric acid treatment, were washed with water, dried, and weighed. They had gained 22 milligrammes, which happens to be just about one-half of the "extra oxygen" reported, as it is when reduced to absolute weight. But this latter quantity is burdened with the observational errors in the determination of the carbonic acid and lithia and the total substance, and consequently affords no safe ground for further conclusions. In an atmosphere of hydrogen neither of the two lithia preparations attacked platinum in the least, even when kept in contact with it for hours at a red heat; but both lost weight to quite an unexpected extent, although the amount of lithia volatilised seemed to have been small in all cases.

In the case of the carbonate the change was more minutely studied. A small platinum crucible, containing 4.219 grms. of carbonate of lithia (recently dehydrated in it at a gentle heat), was placed in the platinum crucible, provided with inlet and outlet tubes, and heated within it for many hours over a good Iserlohn burner,\* while a current of dry hydrogen passed through the apparatus to keep out the air. From time to time the heating was stopped, and the residue examined and weighed. There always was a small sublimate on the inside of the lid of the outer crucible, which was weighed, to be allowed for in the calculation of the loss of volatile matter up to the respective time, and removed before the process was resumed. The corrected loss was very considerable in the earlier periods of heating, but became less and less as the process progressed. It was also noticed that the substance while losing carbonic acid became more and more difficultly fusible. Calculating from the weights of the successive residues and sublimes obtained, and on the assumption that the aggregate (duly corrected) loss was just carbonic acid (lost and not replaced by water), the ultimate residue was very nearly of the composition  $\text{Li}_2\text{O} + \frac{1}{3}\text{CO}_2$ . A determination of the carbonic acid (by the direct method)

\* "Excess" to be taken in reference to the non-lithium presumably present.

† The oxalic acid had been prepared by the decomposition of oxalate of methyl by water, and may have contained methyl-oxalic acid.

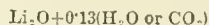
\* A powerful Bunsen, constructed on the Argand principle.



and of the lithia (as  $\text{Li}_2\text{SO}_4$ ) gave the following results:—

Lithia ( $\text{Li}_2\text{O}$ ) .....	88.27+30=2.912
Carbonic acid ( $\text{CO}_2$ ) .....	7.50+44=0.171
Water (by difference).....	4.23+18=0.233
	100.00

These numbers agree with the formula—



Part of this product was utilised for studying its action on platinum in the presence of air. But as a number of days had elapsed before this could be done, and the substance meanwhile had been standing in the crucible (within an exsiccator), we first reheated it in hydrogen gas within the same platinum apparatus as had been used before, with this difference, however, that after thirty minutes' application of an Iserlohn burner the heating was continued for twenty minutes over a gas blow-pipe. Weight of substance operated upon equal 0.4692 grm.; total loss equal 79 milligrammes, which is (by 24 m.grms.) more than the weight of ( $\text{CO}_2 + \text{H}_2\text{O}$ ) calculated for 0.4692 grm. of  $\text{Li}_2\text{O} + 0.13(\text{H}_2\text{O or CO}_2)$ , shewing that the substance while in the exsiccator had absorbed water or carbonic acid. But obviously the substance, by being re-heated, had come still closer in its composition to plain  $\text{Li}_2\text{O}$ . It had assumed a gray colour, and showed only signs of incipient fusion. This gray substance was now heated within the same apparatus, in a current of dry air, a gas blow-pipe supplying the heat. As part of the substance was lost accidentally, the residue was not weighed. It was now of a perfectly white colour, although the empty part of the crucible, through the conjoint action of the air and the vapour of lithia produced, had got badly attacked. This residue again stood in an exsiccator for some days before we had time to analyse it. To remove any absorbed water or carbonic acid we began by re-heating it in hydrogen. 0.347 grm. of the re-heated substance, when subjected to the action of water, dissolved only very slowly, with evolution of very little heat. Without waiting for a complete solution we added excess of sulphuric acid, evaporated to dryness, and weighed the sulphate. It amounted to 1.2459 grm. corresponding to 97.9 per cent. of  $\text{Li}_2\text{O}$  in the substance analysed. To sum up: Carbonate of lithia, when ignited in a platinum crucible in air, attacks the metal, and is itself largely converted into peroxide. A considerable percentage, however, of  $\text{Li}_2\text{CO}_3$  remains as such. When it is heated in hydrogen it does not attack the metal, but gradually loses more and more of its carbonic acid, so that at last almost pure  $\text{Li}_2\text{O}$  is left as an almost infusible solid, which acts only very slowly on water; and consequently is widely different from what the oxides  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$  are supposed to be. (I have no personal experience regarding these myself.) In order to see whether the hydrogen, in reducing the carbonate, acts chemically, perhaps by removing the  $\text{CO}_2$  as  $\text{CO}$  and  $\text{H}_2\text{O}$ , or merely as a supporter of dissociation—i.e., by preventing accumulation of carbonic acid within the crucible's atmosphere—a known weight of carbonate of lithia was heated (within the same platinum apparatus as had served in the hydrogen experiments) in a current of dry nitrogen gas. But as our stock of specially purified carbonate of lithia was exhausted, we operated upon commercial *Lithia carb. puriss.*, as obtained from Trommsdorff. The following weighings were recorded:—

Original substance .....	Grm.
After preliminary dehydration at a gentle heat	0.7227
in nitrogen gas .....	0.7180
Heated over blow-pipe for 15 minutes .....	

The out-going nitrogen, when blown through a flame, coloured it strongly red. Neglecting this loss of  $\text{Li}_2\text{O}$ , and assuming the 0.2398 grms., which the 0.7180 of  $\text{Li}_2\text{CO}_3$  had lost, to be all carbonic acid, it represents 33.40 out of the 59.46 per cent. of  $\text{CO}_2$  present in the original carbonate. The platinum remained unattacked. The heating in nitrogen was resumed, but the experiment miscarried, which is of no consequence, because the result already obtained clearly proved that we have to deal with dissociation pure and simple. I intend to inquire more fully into the behaviour of  $\text{Li}_2\text{CO}_3$  and of  $\text{LiOH}$  when heated in hydrogen or other gases, and to extend my inquiry to the potassium, sodium, and barium compounds.

In conclusion, it is an agreeable duty to me to acknowledge the very able and zealous manner in which I was supported by Mr. Robert Anderson in the execution of the many experiments involved in this investigation.

#### DISCUSSION.

MR. READMAN said: The articles which I have brought with me to-night are a few specimens of various utensils made in malleable nickel, some in pure nickel and others in sheet iron coated with nickel by a process worked out by Dr. Fleitmann, of Iserlohn, Germany. I understand the coated articles are made by rolling thin sheets of malleable nickel on a thin sheet of malleable iron, both previously heated, and having between them a flux of borax. This small cup I have here is coated with 10 per cent. of nickel, the rest being iron. This specimen of a knife is made in pure malleable nickel. The nickel metal itself is usually sold in the form of round cakes, and is extremely crystalline and brittle, as you will see by this specimen. This represents the commercial nickel as it is usually found in the market, differing very much in its properties from the malleable metal manufactured by Dr. Fleitmann. This nickel resembles cast iron much more than malleable iron. The small specimen I have shown, and which I had broken to-day, exhibits tolerably well the crystalline structure of the metal. This specimen of nickel contains 98 per cent. to 99 per cent. metallic nickel. It has been manufactured from the oxide by reduction with charcoal.

*A Meeting of this Section was held in the Rooms, 207, Bath Street, Glasgow, on the evening of the 29th April.*

#### MR. STANFORD PRESIDED.

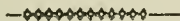
BEFORE calling upon Mr. Tatlock to introduce the discussion on International Methods of Testing and Sampling,

MR. STANFORD said:—It appears to me that there are three points to be considered in the question of testing. First, uniformity in the methods of analysis; then, uniformity in the statement of results; and, lastly, uniformity in the standards employed, and after that comes the great question of sampling. I think that this is rather out of the chemist's province. It is a very important subject, as we all know; but it does seem to me that if the analyst gives a perfectly accurate test of the samples submitted to him, that is really all we can expect from him. As to making this an international question, I would remark only that we have a great deal to do in our own country before we tackle the chemists abroad. When we find that our best analysts admit that even in such a very simple substance as soda ash the results will some-

times disagree to the extent of 3%, we can quite understand that we have a good deal of leeway to make up here.

As to the statement of results, that does appear to me to be a thing which can be carried out by the committee. Those who know the difficulty, for instance, in the statement of the results in mixed salts of potash and soda, know how many variations we find in the statements of different analysts, and how in such a substance as carbonate of potash the statement of the combination of the basis may make great differences in the prices, I fancy it may be necessary to establish a court of appeal; in fact, unless this committee that is proposed has perfect power to decide that some analysts are wrong and others are right there will be very little use in establishing it, and it does seem that we may ultimately require to have some court of appeal to which analyses may be submitted, and the decision of which may be final. We may be a long way from that yet, but it is certainly our duty to try and abolish, as far as lies in us, the great discrepancies in analysts, and wipe out such very objectionable epithets as "high and low chemists."

I am very glad this discussion has to be opened by Mr. Tatlock, who was associated with me in the British Association Committee, and I know it cannot be in better hands. Without further preface I will call upon Mr. Tatlock.



## ON THE PRACTICABILITY OF INTERNATIONALLY ESTABLISHING AND MAINTAINING STANDARD METHODS OF SAMPLING AND ANALYSIS.

BY ROBERT R. TATLOCK, F.R.S.E.; F.I.C.; F.C.S.

It might seem that little or nothing could be added to what has already been elicited, with regard to this important question, in the course of the able discussions which have taken place at the meeting of the London, Liverpool, Manchester, and Newcastle Sections of the Society; but as we have been invited to consider it, and as every locality has its peculiarities, not only in situation, but in respect of the character of the chemical manufactures carried on in it, there appears good reason why we also should record our views in relation to a matter of so much moment both to the manufacturer and the analyst. I presume no time need be wasted in representing the *desirability* of uniform methods, wherever chemistry, as an applied science, is practised and taught; and whatever feeling of doubt may have existed in the minds of many chemists as to the *practicability* of framing and sustaining a universal system, it must now be admitted that the expression of willingness to co-operate, so universally given by all who took part in these debates, has gone far to modify the views of many who hesitated to reply to the original circular of Dr. Lunge, soliciting the views of chemists on these points. If the establishment of a mutually recognised process of sampling and analysing were actually as practicable as it is admittedly desirable, all difficulty would vanish, and all would speedily be in possession of principles and methods of operating which would reduce the discrepancies between the results of chemists to those merely of manipulation. But many who are competent judges have gravely questioned the possibility of an international agreement being accomplished and adhered to, and there is no denying that there is much to be said in support of this view. It is to some of the obstacles which lie in the path of this "consummation so devoutly to be wished," as well as to

some possible means by which some of them may be overcome that I seek to direct your attention, in introducing the subject—**SAMPLING**. There are various reasons why the question of uniform modes of sampling, all-important as it is, should be left in abeyance, until at least some progress has been made in the development of recognised methods of analysis, and these reasons I shall endeavour to adduce:—

1. On an average, an analytical chemist, in practice, does not draw, perhaps, one per cent. of the samples he is called upon to analyse, and is therefore not responsible for them.

2. When samples are systematically drawn, it is customary, at least as far as standard articles such as pyrites, potash salts, etc., are concerned, to divide the samples, and thus obtain duplicates, which are retained under seal, and referred to in the event of discordance between the analyses of different chemists, so that, whatever disagreement there may be, refers, in such a case, to the analysis, and not to the sampling, however imperfectly the latter may have been done, and to whatever extent it misrepresents the goods. In short, the sampling is not called in question unless another sample be taken which is proved to be, or supposed to be different from the first.

3. The literature on the subject, if any exists, is practically *nil*, as compared with what is at the command of the chemist in subjects directly bearing on technical analysis.

4. Both raw materials and manufactured products exist under such a variety of conditions, and under circumstances so varied, that only very general directions could be given, which would not be of use only to a novice. More harm than good might result from laying down specific instructions, which would require to be followed by an experienced sampler, or which would interfere with his judgment, particularly under special conditions over which he had no control.

It would be useless to deny, that if the object of sampling and analysing goods is to arrive at an exact estimate of their strength or composition, and consequently of their value, the one operation is of equal importance with the other, for it is plain, that while great care in the selection and carrying out of analytical methods may save the reputation of the chemist, it will not save the pocket of the manufacturer or consumer, whichever he may be, who sells or buys on an analysis of a sample which does not accurately represent his goods. From the above considerations, however, I am of opinion, that for the present at least, sampling had better be left to the discretion and experience of individuals, particularly as it is a process which depends more upon the exercise of common sense, sound judgment and experience, than the observation of any rules, however carefully thought out and scientifically elaborated. I am not desirous of pressing this view too strongly, but in support of it I shall just quote three examples with the object of illustrating what I mean by the difficulties attending a prescribed system of sampling:—

Example A.—A delivery of 50 boxes of Italian olive oil soap had to be sampled; the goods being sold on a basis of 62 per cent. of fatty acids. The quality of the *total contents* of each box may be different. The proportion of the valuable ingredient cannot be the same in every bar in a given box, from the fact that some of the bars have only their ends exposed to the outside, others their ends and one side, and a third series their ends and two sides, while a fourth may be completely interned. Then again the bars selected for analysis, for the same reasons, are also in different conditions of dryness, and the sampling of each bar, by the analyst, for his working sample, becomes a matter for grave consideration. The problem is—What proportion of boxes are to be opened, from what



positions are the bars to be selected, and in what way are the selected bars to be punched out so as to give an accurate average for analysis? Each one per cent. of fatty acids represents about £1 12s. 6d. on every £100 value; but probably any hard and fast method of sampling would be completely upset by the adoption of a different form or size of box.

Example B.—A few thousand boxes of Japanese fish oil have to be sampled; many of the boxes have from a mere film to a quarter of an inch of water at the bottom, and the oil is of such consistence that it will not run in a syphon, nor will it either incorporate with the water on being stirred, or mix with it on inverting the box. The ordinary slide tube for oil-sampling is useless, as the metal plate which forms its bottom is as thick as the depth of the water. The question to be settled is—How many boxes are to be taken, and how is an average sample of the whole contents of the selected boxes to be obtained?

Example C.—A cargo of 1000 to 2000 tons of Spanish iron ore has to be sampled. The goods have every degree of size, from fine dust up to blocks weighing several cwt., every shade of quality from ore of 60 per cent. iron down to rocks, stones and clay, and in every condition of moisture, from absolute dryness to 20 per cent. of water. Is a man, single-handed—his only tool a hammer, his only guide an international code of instructions—equal to the task of taking a sample which shall fairly represent these goods? Or let us suppose that the goods pass on to an iron work and are there sampled—where the assistance of men with sledge hammers, buckets, barrows, pan-mills, and all the paraphernalia of the practical sampler can be had. In the meantime, however, two things have happened to the ore—it has been weighed for invoice purposes, and afterwards it has been deluged with rain to an unknown extent, thereby increasing its weight from, say from 1000 to 1050 tons, and so reducing its original percentage of iron from, say 50 to 47·62, thus compelling the vendor to sell on dry weights and wet sampling, and in so doing to sustain a loss of at least £40 on such a cargo, of which there are probably twenty at least in his contract.

Will any international combination attempt to lay down rules, which to an experienced sampler will serve him better, under all circumstances, than his own judgment? It must always be borne in mind that any universal method must be a practicable one; and must not involve immoderate trouble and expense—the object being a purely commercial one.

ANALYSIS. It is far from me to attempt to defend errors in the work of analytical chemists, or to attempt to justify any discrepancies which may occur between their results; but it seems to me that such confessions of incapacity as we hear made frequently are not warranted by facts; and I do not altogether approve of the "*mea culpa, mea culpa, mea maxima culpa*" style in which some chemists are wont to air their little imperfections; and especially before the public, who, however discerning, cannot be expected either to know or appreciate the agony of thought and work which a chemist has often to go through, and the pecuniary loss which he frequently sustains, in endeavouring to get an honest result. If anyone has not had experience of this, let him accept a commission to determine accurately the gold and silver in some such ore as that of Tharsis or Rio-Tinto; and, having got results sufficiently correct to bear comparison with those obtained in the extraction of these metals on a working scale, let him attempt to charge a fee at a very moderate rate, say one shilling per hour, based upon the time he has expended, and I think the result will be that his views in this respect will coincide with mine a little more closely than before.

It is all very well to select the comparatively few instances in which chemists' results disagree; but who will bring under our notice the countless instances in which they harmonise and coincide in a manner which is, to say the least, surprising even to the chemists themselves. We hear much also from other interested parties with regard to discrepancies in results, and although I am very loth to refer to my own practice I shall quote only one example to illustrate what is expected from chemists. Within the last week a London firm who have got many hundreds of samples of potash salts analysed in their day, wrote to me demanding to know how there could be such a discrepancy as 25 per cent. of sulphate of potash in my analyses of two duplicate samples of that article? Of course, I replied that the difference was quite within the permissible limits of error. The fact is, that buyers and sellers, as well as manufacturers, are getting more "keen" than formerly, by reason of small profits as a necessary consequence of over-production and competition; and the probability is, that even with more refined and more uniform methods we shall hear as much of chemists' errors as ever—perhaps even more—for, as we go on refining advantage will be taken of it, until what is now accepted as a permissible difference will be regarded as a totally inexcusable discrepancy. The truth is, that the chemist is not unfrequently made to answer for the sins of the sampler, of the railway companies, and even of the "clerk of the weather." A buyer of caustic potash draws a sample of the article from a drum, and has it analysed; the result is reported to the seller, who indignantly repudiates the analysis, goes or sends to see another sample drawn—from the other end of the drum, of course—and has it analysed, with the usual conflicting result. Or a couple of thousand bags of muriate of potash are sampled at the German factories, put on the waggons, forwarded to Hamburg, transferred during wet weather to the ship, brought to Leith, unshipped, placed on open wagons, and carried, say to Glasgow, where they are again sampled under an umbrella, and an analysis is made. The result naturally differs from the German, perhaps by about 1 per cent.—the difference being chiefly accounted for by the moisture—accounted for, at least as far as the chemist on this side is concerned; but his efforts to convince the "agent" who has bought the goods on German and sold them on English analysis are utterly futile, and the "hue and cry" is got up—"another gross discrepancy."

But apart from all this there is no doubt that grave differences exist, for which the chemist is truly responsible, either by his method or his work, and I believe this very frequently arises from the impossibility of a chemist being a specialist in every branch. Have we not all been largely benefited by the advice and assistance of our professional brethren who were specialists in some particular department with which we were not so familiar, as well as of manufacturers and their technical managers and chemists who had brought special methods of analysis to a degree of perfection hardly dreamt of either by the commercial analyst or the scientific chemist. This very fact should be sufficient to convince us that much good might be done by a combination, with the object of making all knowledge of analytical methods, at any rate, common property. Would it be too much to expect that such an institution would accomplish the following results:—

1. To lay down standard methods of analysis for some of the leading commercial products to begin with, extending the list as opportunity offered.

2. To do away with trade customs based on erroneous principles, such as the 32 equivalent for soda,

the unscientific saltpetre "refraction" process, the barbarous Cornish assay, etc.

3. To establish uniform modes of expression in stating the analysis of the more commonly occurring commercial products.

4. To do away with the practice of employing arbitrary scales in instruments used for measuring temperature, density of fluids, strength of chemical products, etc., and to place those on a basis already universally understood.

5. To appoint a "court of appeal," in order to settle cases of disputed analysis, or at any rate to recommend specialists or experts who were considered competent to act, and who would not charge special fees for their services.

It is quite true that many difficulties lie in the way, and it may be as well, even at this stage, to look some of these in the face.

It has been argued, and not without reason, that such a combination would be too dictatorial, and that it would tend to hinder the free and natural development of analytical processes, bar the progress of individual enterprise in that respect, and act as a drag on improvement generally. That would be the tendency, probably, if the rules, or the action, of the Association were too rigid, or if an attempt were made to fix hard and fast lines without making proper provision for the natural growth and expansion of improved methods. It seems to me that all objection on this score might be obviated by members communicating any alterations which they had found by experience to be desirable, with the experiments upon which their suggestions were based, in some recognised journal, or in some magazine devoted to that and the other objects of the Union—such proposals to be considered from time to time by an international conference consisting of small committees appointed by the entire body of members in each country.

The Association would, of course, keep always in view its chief *raison d'être*, which would be that of testing, providing, and improving from time to time, not time-consuming and costly methods of analysis, but practicable ones, such as could be employed by the chemist and the manufacturer without such an expenditure of time and money as would render the work valueless for commercial purposes. It would also urge the adoption of uniform modes of expressing results, which for some time would cause a good deal of extra trouble on account of their not being understood by the non-chemist, who would require to be educated to them gradually. It would also, perhaps, publish series of tables useful to the analyst, including analyses of natural products, for reference, such as we have in the admirable special German Works on Agricultural Chemistry.

It would not be necessary to have absolutely rigid scientific methods, although it would be extremely desirable to have them as perfectly as possible, on a scientific basis. They would thus bear the same relation to strictly scientific processes, as the individuals of a set of weights which agree among themselves, but which were not based upon any recognised unit, would have to a similar set based on a determinate unit. It has been hinted, in the course of these discussions, that only such portions of ingredients should be determined as can be profitably extracted on the large scale. I must say I have no sympathy with such a view, and I trust we are far from seeing the day when it will be adopted. Such a course would, in many cases, open a flood-gate for discussion. A chemist has nothing whatever to do, as far as stating the percentage of any given constituent of an article is concerned, with how much of it can or cannot be extracted with profit, as circumstances in these respects are constantly changing. His duty is clearly to represent the

entire amount of the ingredient in the sample, as he received it, and to make any explanation regarding it which he considers necessary. How would it suit to report only 28 per cent. of zinc in a sample of calamine in which 35 per cent. was found by analysis, because the chemist held an opinion that not more than the smaller proportion could be economically extracted? It would be as reasonable to add to the amount of iron found in an iron ore, 10 per cent. of its weight, on the ground that 100 parts of the manufactured pig iron contained only 90 parts of pure iron, or thereby.

Fears have been expressed that the recommendations of an international association would not be universally acted upon; but it is not to be expected that in all cases they would. Trades Unions, combinations, and syndicates, who have monopolies, will not always accept reasonable conditions, if they can enforce others which contribute more to their pecuniary advantage. It may be interesting—certainly amusing—if not instructive, to take a glance at the tactics of one such body—the Syndicate Manufacturers of Muriate of Potash in North Germany. The following is an excerpt from a translation of the "conditions of sale of the 'Muriate' Manufacturers' Syndicate in Stassfurt":—

"The Analyses will, in first instance, be made by the factories, and afterwards by the sworn chemists attached to the official laboratory of the Syndicate. The latter's mean result will be taken as final. In case of the two official tests differing in their results, the one from the other, to the extent of 0.3 per cent., the samples will again be submitted to another test." It is worthy of note, that there is no appeal even to any "Haupt Analytiker" of the fatherland; and the joke of swearing in samplers and chemists who are responsible only to, and are paid by, the "Syndicate," is rather a good one. It may be worth while to examine the character of certain analyses which are issued under the auspices of this association; and here again I must apologise for again referring to my own practice. I take the liberty of quoting the very last one which has come under my observation, and of relating the circumstances in connection with it, which have transpired within the last week. On the 12th of the present month I received a sample of 1680 bags muriate of potash, which had been purchased by a well-known firm in this town, the delivery representing upwards of £1,500, and constituting only one under a contract. The gentleman who sold these goods was, unfortunately for him, obliged to buy them on Syndicate analysis, and to sell them on mine. He accordingly furnished me, at the same time, with a copy of the former, which is reproduced in the first column of the undernoted three analyses. My analysis of the sample appears in the second column. It was duly sent in, and elicited a note from my client, in which he said: "I am surprised at the result of your analysis again. The German analysis has been done at least three times, as I have shewn you, on Fresenius's principles, and according to your own treatise there should not be greater difference than  $\frac{1}{2}$  per cent. between him and you, and you made  $1\frac{1}{2}$  per cent. It is really quite impossible, under the circumstances, to sell on your analysis." I replied to the effect that even if I had returned the German figures, I should not have had room for the other impurities, as the analyses would then have added to considerably over 100 parts; and suggesting at the same time, that he should send one of the samples which had been simultaneously drawn and sealed by his and by the buyer's representative, and which had never been in my hands, to Professor Dittmar, and in the event of his coming nearer to the German results than mine, I should pay all expenses; but if *vice versa*, he should do so,



This he generously accepted, and Prof. Dittmar's analysis appears in the third column. The reference to Fresenius will not be overlooked, but it is one thing to quote Caesar's authority, and another thing to "appeal unto" it; and I have generally found that any proposal on my part to refer a disputed analysis to that really excellent authority has resulted in the subsidence of the complainant into silence, even when the offer was made to defray costs in the event of the result going against me. The following are the three analyses referred to:—

	Syndicate.	Tatlock.	Dittmar.
Chloride of Potassium ....	98.90	97.52	97.65
Chloride of Sodium .....	35	89	89
Sulphate of Lime .....	not given	20	06
Chloride of Calcium .....	"	—	01
Chloride of Magnesium .....	"	35	12
Insoluble .....	"	21	14
Water .....	70	81	100
	99.95	99.98	99.87

A great deal has been said with regard to differences alleged to exist between the results of analyses made by the method recommended by Fresenius, and those got by a slight modification of it, which I have been in the habit of using with advantage for the last quarter of a century. As one of the objects of our meetings is to have the facts relating to such important matters laid open to criticism, I take this opportunity of laying before you the results of all the analyses, as far as they are known to me, that have been made by the veteran analyst of Wiesbaden, of duplicate samples, the originals of which I had previously tested during my entire practice. In all the cases my analyses were disputed on the ground that they were far below the results obtained at Strassfurt, with samples of the same lots of goods, and consequently as far below the truth. The figures represent the percentage of chloride of potassium found in each case:—

	Strassfurt.	Tatlock.	Fresenius.
1.		88.65	88.86
2.	96.30	93.85	95.26
3.	97.90	96.36	97.54
4.	96.80	96.30	96.26

It will thus be apparent that while the referee is in two instances above me, he is in other two lower—his mean result being 18 *over* mine, while in every case he was much under the Strassfurt figures—his mean result being 64 less than that of the latter.

My chief object in representing these facts is to point out that it is unlikely that trades unions will, in all instances, adopt the processes prescribed by an international association, but there is all the more reason why one should be instituted, in order to counterbalance the influence of these, as otherwise special industries may take steps to dictate methods uncontrolled by any recognized authority, and naturally of a character most suited to their own interests. Can we have these industries laying down the law, and dictating to and leading the chemists in these matters, as the Association of German Manure Makers and the Alkali trade in Germany have already done?

One great advantage that would flow from an international arrangement would be the abolition of customs in stating analyses, which are pernicious in a high degree. I quote but one example. Having heard that certain well-known analysts on the Continent were in the habit of representing as carbonate all the phosphate or potash present in carbonate of potash, and which might amount to 5 per cent., and having been frequently troubled by discrepancies between their analyses and my own being pointed out

to me, I wrote to two of these gentlemen, asking whether I had been correctly informed. One replied:—"Je represente, sous forme de carbonate toute la potasse qui n'est pas combinée avec le chlor et l'acide sulfurique. C'est l'usage ici et en Allemagne d'opérer ainsi. On ne tient compte de phosphate de potasse que par suite de conventions expresses entre vendeurs et acheteurs. Je vous le repete, c'est l'usage commercial, et si j'agissais autrement, je me trouverons tous les jours au désaccord avec mes confrères."

Another admits the practice, and says:—"Je regrette de me trouver aujourd'hui en contradiction avec vous, mais espere que notre Bourse se decidera à adopter une méthode à la hauteur du moment, et nous placant sur le terrain de la science, et non sur cela des coutumes."

One fear is that such an Association would move too fast, by dictating alterations and improvements which would not meet with the sanction of many of its members; and this would not be desirable even in the event of the dissatisfaction of a considerable minority. Sudden changes would also tell rather hardly upon existing contracts, particularly those that had a considerable period to run; but surely this could be arranged, and chemists who were working under such, could retain their custom till the expiry of them, giving their clients due notice of the necessary alteration.

Above all things it would be desirable to secure the assistance of chemical manufacturers and manufacturing companies whose technical managers and chemists are specialists in their own department. What professional chemist in active practice does not remember with gratitude the aid he has frequently received from such friends who have elaborated methods, which, for accuracy, speed and cheapness, left nothing to be desired, and whose systems of working and checking results I have not seen surpassed, or in some cases even equalled, by those of any professional analyst.

Of course the action of the Association would not in any way interfere with the application of special methods which manufacturers or chemists for the sake of speed, or for other reasons, might employ for their own satisfaction. The prescribed methods would be intended only for mercantile purposes, or where the results of different chemists were liable to be compared and challenged.

Resolutions have already been adopted by more than one section of the Society, which plainly imply that there is a strong and widespread belief among its members that co-operation, with the object of bringing about an international agreement, is practicable as well as desirable; and, believing that many of our local members participate in this feeling, I have much pleasure in proposing the following resolution for the consideration of the meeting. It is substantially the one which was moved by Mr. John Pattinson at the London and the Newcastle meetings, and unanimously carried:—"That the Glasgow and West of Scotland Section recommends the General Council to appoint from the members of the Society a committee, composed of chemical manufacturers, consumers and analysts, whose duty it shall be to determine whether it is desirable to endeavour to obtain national or international agreement on chemical questions affecting the value of commercial products, and to report to a future meeting of the Society."

Mr. MACTEAR said: In rising to second the resolution which Mr. Tatlock has so well put before you, I would do so without saying very much in support of it. I think the subject is one which, while we may discuss to a large extent here, can only be

thrashed out by such a committee being formed. I think that it will be an exceedingly good thing, which would result in good and good only, to have that committee, and to have them exercise their powers with intelligence upon the subject for some little time before we have a complete discussion or make up our minds on the subject. I think there are many cases in which it is absolutely impossible that methods can be laid down which would be adopted in all circumstances. There are many points—very many points—where an investigation would lead to the wiping out of very peculiar customs, very fallacious figures, and to the establishment of standards which we could go upon and which might be trusted. If we take for instance the very simplest thing of all,—the strength of oil of vitriol—I daresay that at the present moment there is no manufacturer of oil of vitriol, or any consumer that I know, who is perfectly agreed upon what oil of vitriol is, and yet it is the foundation of our whole chemical industry. It is no uncommon thing to be asked to tender for oil of vitriol of 170°. Oil of vitriol of 170° Twaddell cannot be made pure. You are asked to tender for oil of vitriol of 170° free of iron lead and other impurities, and yet such a thing is not attainable. On the other hand, very few of us know what oil of vitriol is, and so far is the case that the American manufacturers, some two years ago, found themselves in such difficulty to decide what oil of vitriol was that they formed a committee, who went through a very large amount of work and determined what a Baumé hydrometer should be, what degrees oil of vitriol ought to be, and have drawn up a table of the strength of sulphuric acid, which is now in general use in the United States, and which, I think, is the most perfect table of sulphuric acid that we have. A similar piece of work might easily be done by such a committee as you propose to have by this resolution; but when you come to deal with the question of analysis the difficulties that come in are exceedingly great, and one of the difficulties that I see, and which I doubt is impossible to be overcome, is that any method which may be laid down will give very different results according to how it is carried out by different hands. I have had a considerable amount of experience, and a considerable amount of work in connection with the testing of the escapes of nitrous compounds from vitriol chambers. The estimation of the nitrous compounds has been an exceedingly difficult one, and I ultimately adopted a system of distillation with zinc and iron and soda which, in my hands, and in the hands of my assistants, has given important results which are quite as perfect as almost any analysis in a technical work would require to be. On the other hand, that process in some other hands has been condemned right and left. It has been said that it was impossible to get results which are at all steady or trustworthy by such a process, and yet the whole difference lies in the carrying out of the process, and as to whether evaporation is carried up to a certain point, or not carried so far. In a case of that sort, supposing that an international committee decided that such a process was to be adopted, it would by no means avoid the danger that great differences might arise in the different hands employing the process. There is another point which is difficult to deal with—that is trade custom. There have been a good many discussions in my time regarding common systems of testing, and at all of these discussions the particular question of the testing of soda ash has been brought up as the one flagrant example of what trade custom is. Now, I think this testing of soda ash was very ably put before a meeting in Newcastle, by Mr. Pattison. Still it is not understood by most

people. The test which is called the English test for soda ash, and which equivalent of soda is taken as 32 instead of 31 and a decimal, was established when 32 was believed to be the proper equivalent of soda. It would, therefore, be very unfair to the manufacturer of soda, and here I speak more particularly for my fellow manufacturers of soda ash in England, because in Scotland the system of using this test was abandoned at a very early date, and has been for many years the 31 equivalent, but I consider it would be an extremely great hardship, that because refined means of testing, and later investigations, had shewn that the chemist was wrong in his original equivalent of 32, the trade custom, upon which the manufacturer has been selling and the purchaser buying, should be altered. The purchaser is not now paying more than he did formerly for a ton of soda ash of a given strength when tested on the same basis, and all these commercial tests are really relative tests. If you change these relative tests, and reduce the equivalent to 31, you thereby make a change which would reduce the value—whichever way you like to put it—and mulct the manufacturer in somewhere about 3s. per ton. It is all very well for sentiment to say it ought to be correct, the manufacturer may recoup himself by charging a higher price, but anyone who is a manufacturer knows it is absolutely impossible to charge a higher price. The answer you would get would be, "Your test has been wrong; you don't expect me to pay for your error." And to a certain extent, no doubt, the customer would be correct. I think these trade customs will not be abolished by any such Association; and if such Association interferes with them, the probability is that the advantages of it would, to a great extent, be lost, because the recommendations would not be adopted. Still, these are points for such a committee to take up and thrash out, and I have no doubt in many cases, by putting before the consumer a statement or table, such as Mr. Pattison drew up some years ago, in regard to soda ash and bleaching powder, they would at once see what the relative differences were, and they would be able to calculate their price accordingly, and not pay for what they were not getting. There are many systems of analysis, and of testing, which have been adopted, more especially in technical works for the special circumstances of the case which one has in hand; and while I think that a general text-book, which might be applied to the ordinary cases of analysis which are occurring every day, would run every chance of being adopted by the majority of technical men, I doubt extremely if any of them would adopt these systems to the exclusion of all others. Mr. Tatlock has put very well before us to-night the extreme difficulty of dealing with the sampling question. I sympathise with him most heartily in the question of sampling. It is far more difficult to procure accurate samples, to put before the analysis, than to get a true and accurate analysis of the sample once you have obtained it. There is many and many a time that a sample is taken by a man who has not the remotest idea that it is necessary to mix the sample thoroughly, and to fill his sample bottles equally with what will really be a representative portion of his material. To some men, who are entrusted to take samples, it is quite sufficient that he takes from the cask or bottle—or whatever it may be—two or three bottle fulls, which are sent off as being a representative sample. In one case which I happened to know of personally, where a sample was sent to an analyst in Scotland, it was a sample of soda ash. The man who had taken the sample had left a considerable number of chips, which had come off the hole he bored in the cask,



The first analysis that was made of the sample contained one large chip, and turned out very low, and when the test was objected to by the person in Liverpool, the analyst's reply was that he remembered seeing this chip, but as it was in the sample it was not his business to take it out, and in making another test of the sample without chips he found it up to the test and according to the bargain. In sampling other things, such as pyrites, you have great difficulties, more especially if you have cargoes in which the pyrites crumbles down, and more especially as to the copper in it, because the small and large often vary. And, in speaking of pyrites, I would like to point out, in relation to what Mr. Tatlock said about chemists having no business to report what he considered to be the actual value of the material, and in regard to what could be taken out of it, that consumers of pyrites are sometimes—quite unintentionally I believe—placed at considerable disadvantage. The bargain that is made in regard to pyrites is so much per unit of sulphur, and you sometimes get an ore that contains one or even up to  $2\frac{1}{2}$  per cent. of sulphate of copper, or sulphate of iron, which is utterly useless to the manufacturer of sulphuric acid, but for which he has to pay as available sulphur present in the ore. These, and many other points, would all be thoroughly discussed if we had representative men on the said committee, and I have, therefore, great pleasure in seconding the resolution.

Mr. T. L. PATTERSON said: I would very much like to hear the opinion of some of the gentlemen present who are engaged in some of the large chemical works. It is a long time since I had anything to do with potash salts and mineral phosphates, but I may say that we have an Association in the sugar trade, intended to meet some of the difficulties suggested by Mr. Tatlock, and which has been referred to by my friend, Mr. Newlands, in the London Section. The Association consists of sellers on the Continent, brokers and buyers here. They have their own chemist, whose business it is to analyse samples sent to him by the Association. In the first place, samples of sugar are taken on the Continent, and analysed by the chemist there—that is by the sellers' chemist. His analysis is sent to this country to the broker. Sugar when it arrives here, is taken charge of, sampled, and weighed on the quay by the Association; the buyer pays for the weight that passes the scales. The seller and buyer have nothing to do with the sampling, nor with the weighing. The whole thing is done by a representative of this Association. The sample taken on the quay is thoroughly mixed and divided into three portions; one portion is given to the buyer, and the other two are retained by the Association. The buyer gets his sample analysed by his own chemist, and when finished, he exchanges analysis with the broker. If the two analyses do not differ by  $\frac{1}{2}$  per cent.—in case of sugars sold on a basis of 88 per cent. net—the mean of the sellers' and buyers' analysis is taken, and the sugar is paid for on that mean. Should, however, the difference exceed half per cent., the Association causes one of the two remaining samples in their possession to be analysed by their chemist. The three analyses are now compared—seller's, buyer's, and Association's—and the sugar is paid for on the mean of the two which come nearest. I don't think there is anything to hinder such an Association being established in connection with other chemical products. I think if the Association in Germany, to which Mr. Tatlock has referred, were combined with a similar one in this country, and the combined Associations appointed a neutral responsible party to sample the material as landed on our quays—such sample to be sent to a similarly appointed chemist, in the event of a difference between buyer

and seller, whose decision would be final—I don't think there would be any difficulty at all in arriving at a true solution of the question. That would do very well with potash salts, and all materials that are in a crystalline or granular condition, but when we consider the sampling of pyrites to which Mr. Mactear has referred, that would be much more difficult. I don't know how it would do in that case; perhaps some gentleman who is acquainted with it can say. In the question of iron ore, or mineral phosphates, it is very difficult indeed to arrive at proper means of sampling them. I might say with regard to sugar, that we had very great difficulty at first in getting this Association started. Formerly we used to find that all the sugar that came from the Continent weighed about 127 per cent. less than when it left. We also found the analysis about one per cent. (1 per cent.) less than what it was on the Continent. This was believed to be due to the sample absorbing moisture from the time it was sampled until it got into the chemist's hands. For that reason we always made allowance for excess of moisture, and if, after making that allowance, the sample came within a half per cent. of the foreign analyses, the sugar was passed. By-and-bye it was found that the foreign analyses showed very little water, and that even after making this allowance the analyses did not come right; and some knowing ones on the Continent, on investigating the matter, found that some unscrupulous sellers were drying the samples before sending them to the chemist, while we had still to make water allowances over here. These are some of the reasons which, to the starting of the Association, and since its inauguration, I don't think there are more than probably ten per cent. (10 per cent.) of the samples that require to be sent to neutral chemists. The samples that are analysed on the Continent agree with the samples analysed here, and the weight on the Continent is generally a little less than here.

Professor MILLS said: I have heard the discussion which has taken place to-night with very considerable interest, and perhaps I may refer to a singular point that has struck me; that is, with regard to actual (tests) analysis. All chemists in their daily work are constantly finding that they are making errors—what Mr. Tatlock calls permissible errors—errors which may be excused. I think therefore it would be a very great advantage to this enquiry, which is about to be commenced, if all such errors were duly tabulated. Supposing, for example, that Mr. Tatlock were to take a particular sample of potash salt, and make several determinations in his laboratory of the amount of potassium in this particular sample, and then ascertain the average departure from the mean result. And supposing that all analysts, who are specialists, were to do so, we should then, at any rate, have a very valuable starting point or basis to go upon in ascertaining the amount of permissible error in ordinary analysis. This, I think, should be a preliminary enquiry. I certainly think very considerable stress should be laid upon the method of sampling. At present we have no theory of sampling, and samplers, as far as I know, have said nothing as to their methods of sampling, or the principle on which they go. The truth is, that sampling is a matter of practical mathematics; in fact, a branch of the theory of mean value, and we cannot expect persons who sample in the ordinary way to be acquainted with this. At present we cannot do such a thing as properly sample a pig of iron, as we do not know what is the exact position of the surface of mean composition within the pig. In every case we are hindered very much by the form of the vessel in which the original matter is contained. A cask of brown sugar, for instance, is not

convenient for uniform sampling. Two or three other things have struck me. For example, in a cask of brandy, the brandy has a different composition in the wood from what it has in the body of the spirit. Then in some cases climate comes into play, and we have in the case of water supply a very grave difficulty, that it will take eleven years to ascertain what the average nature of the supply is. So from first to last chemists are very much troubled with the mere fact of sampling, and I think it ought to be one of the regulations, as far as possible, to get samplers to speak out. If they do that, they will materially assist this very difficult and grave question.

Dr. MILNE said : I have only to express very great pleasure with what has been said to-night, and to say that I think it is a step in the right direction. I think it is a very good point as regards our own Society here, in contrast to what is being done in that way by other societies, such as the Institute of Chemistry. I understand that one of the original objects of that institute was something similar to what has been discussed to-night, namely, that there might be a careful supervision of analytical processes and various other matters. That, however, has fallen entirely out of sight ; at least I have not been able to see any result that has followed. I can quite sympathise with Mr. Tatlock regarding sampling, and I would refer to one case that happened not very long ago in my own experience. I had occasion to take a sample of iron ore on board a steamer. I went down personally to do so, and one could not but notice how utterly useless was my going down at all. In the first place, there was no one there having any authority in the matter. I explained what my business was, of course, and tried to get assistance ; but about the most important thing I had to do was keeping clear of the descending buckets ; and as for getting any of the crew to give me assistance, that was almost next to impossible. The greatest difficulty I had was in getting them to understand that I wanted to take an *average* sample of this load of iron ore. Their idea of sampling was to pick up one or two pieces, and throw them at me. As an average sample, this was of no use, and I felt all the time—although I had taken the sample—that, as a representative sample of the cargo, it was simply worthless ; and I think this must have occurred to anyone else who had done the same thing. As regards the difficulties of having any fixed or definite processes, I quite agree with what has been said as to the obstacles in the way of getting everyone to adopt any uniform mode of analysis ; but I think there is at least one good that might result from such an enquiry as has been proposed, and that is, that although we might not be able to get everyone to adopt our system of analysis, we might at least be able to convince some people that their systems were erroneous. If we do that, it might be a step in the right direction ; and after that, we might be able to get them to adopt what we consider the correct mode of analysis. As the subject has been so fully gone into, I have only to say that I agree very heartily indeed with Mr. Tatlock's resolution, and I certainly hope that good results will follow.

Mr. John MILLER said : I have listened to what Mr. Tatlock has said regarding sampling, and to the objections he has raised regarding the latter, and I should like him to explain to me how it is that smelters find so little difficulty with their samples, they being in the habit of buying, not on their own but on the sample drawn by the broker in Liverpool, and, judging from the biddings, those samples are always just and proportionate. If it is possible to sample rich argent ores it is likewise possible to sample a substance like potash, and I think when a difference

does arise, it is due to the fault of the analyst, or the method employed by him and not to the sample, assuming the latter had been drawn by one whose business is to sample. Mr. Tatlock shows analyses of the same potash salt, made by himself and other chemists, and I notice there is a difference in one case of 1 to 1½%. Such a difference of 1% in the precious metals would simply mean ruin to buyer or seller, and in no instance, in my experience at least, has it occurred. If it is perfectly possible to sample with accuracy gold and silver ores, why cannot the same be done with potash ? I had an instance the other day wherein material was sampled in our work the value of which was about £3,000 per ton and when assays were adjusted between buyer and seller there was only a difference of 1-cwt. or 2-qtrs. of gold in a ton. No doubt differences will arise and sometimes the correctness of samples will be doubted, but when the "professional" sampler resamples the material, the first sample is generally confirmed by the second. In other instances I have found the assays of a large number of lots differed always in a certain proportion, and on investigation it was found that the difference was fully accounted for by the methods employed by the two analysts. Prof. Mills has referred to the difficulty attending the sampling of a bar of iron. Such difficulty does not apply to lead, the biddings of various buyers for the same lots silver lead being always remarkably close upon one another. Mr. Tatlock objects to a chemist, in reporting, say on a zinc ore, not the percentage he had found, but the percentage capable of being extracted. This is simply a question of convenience ; if he did not do it the buyer would. One chemist finds, say 45% lead, and another reports 40%, leaving a margin of 5% for loss in smelting. I fail to see there is any objection to this. In conclusion, the difficulties supposed to lie in sampling bolts of various kinds do not apply to the precious metals ; in the latter case it is perfectly easy to get a perfectly just sample, and, admitting this, why is it not possible in other things ? When a sample has been drawn by one whose business is to sample, and on testing it two analysts differ, then it is almost certain the difference is due to the analyst or method employed by him and not to the sample.

Mr. CHRYSTAL said : I think that all analyses of minerals, or such materials as referred to by Mr. Tatlock, should be done in the dry state. A great many of the differences arising over the various analyses of iron ores, &c., were frequently due to differences in the moisture. Take a cargo of iron ore from Spain, which is a much warmer and drier country than this, arriving at our general terminus being discharged into trucks in a pour of rain, and being subjected to two or three days, rain during transit to the iron works, it arrives there, with, say, 5 per cent. moisture, while originally it may only have contained 1 per cent., this of course must lower the percentage of iron, but it increases the weight. I think an arrangement could be made, as is actually being done at present, to fix a limit or standard for moisture, say the 1 per cent., calculate the iron found in the sample up to this percentage of moisture, and deduct from the weight of the cargo on arrival the excess of weight due to the excess of moisture. Even in the case of muriate of potash I have found many of the differences due entirely to a difference in the moisture found in the sample ; where the analyses are calculated to the same moisture, or to the dry state, the differences vanish. However carefully samples have been taken and mixed when sent in for analysis they come out with a difference of ½ to ¾ per cent. One person dries the sample taken direct from the bottle, while another empties out the whole bottle, grinds it, leaves it per-



haps lying for some time in a warm laboratory, and then weighs out the quantity for moisture; I think this is one point where a great many differences come in.

Mr. JOHN HUTCHINSON said: The deliberations of a committee, such as Mr. Tatlock proposes, could be easily brought before the notice of commercial men and analysts, and a regular system of sampling as far as it is possible to have a regular system, agreed upon. But there is still the difficulty with private individuals who may find a necessity for sending samples for analysis. To show that most erroneous ideas exist, both as to the quantity required and the manner of drawing the sample, I will quote two examples from my own experience. In one case when a complete analysis of a water was required an ounce phial full was sent for the purpose. The other incident I refer to was in connection with a sample of manure upon a heap of which the farmer who sent the sample had thrown some liquid ammonia, which he happened to have in his possession, and for which he had no use; quite forgetting he had done so, he drew the sample from that portion drenched with ammonia. On analysing the sample it was evident there was something wrong, and on communicating with the sender of the sample I learned the above facts. While these are trifling incidents I bring them before your notice in order to suggest to the committee the advisability of compiling instructions as to the best manner of sampling, which could be forwarded to private parties who wish to send samples for analysis. I believe that the great majority of discrepancies in analysis are due to the sampling. In one case where I have had experience for about 10 or 12 years in analysing samples of manure for a firm whose samples are carefully drawn, mixed, and portions of the mixture sent to three different chemists no case of discrepancy has arisen, nor is there any difficulty with the results.

Mr. BIGGART said: As a representative analyst of associations which are actually in existence and doing good work (I refer to the Greenock and Lancashire Beetroot Sugar Associations), I may be allowed to add a few remarks supplementary to what Mr. Paterson has already said. I would say that while Mr. Paterson intimately represented the practical or working side of the sugar industry, regarding which I shall consequently be silent, I will, as an analyst, confine my remarks to the analytical work in its various aspects undertaken by the Associations. In the course of a season I pass many sugar samples through my hands, and I thoroughly sympathise with the remarks that have been made regarding sampling and the difficulties which arise in connection therewith. For example, in some cases, particularly in those of the lower or second product sugars, very great differences may occur if the average sample be not very carefully mixed. I know of such a case in which two samples, one taken from the top, and the other taken from the bottom of a heap, lying on a sample table, were sent to another analyst and myself respectively. The difference in the result of the analyses was found to be about 2 per cent. Unable to understand such a discrepancy, our samples were exchanged, when the original results found by each were confirmed, thus shewing that the difference was in the samples, and that it clearly arose from insufficient mixing. With due care, however, we find little or no trouble in this direction. As regards calculating results to a dry sample. This, in some commodities at least, seems to be quite unnecessary. Sugar, for example, is bought on the basis of the weight that passes over the scale, and the samples are secured at the time it is being weighed. It follows almost as a necessary consequence, and is often observed, that those lots of sugar which shew an increase of moisture

on analysis, also shew an increase on the invoice weight, so that the loss on analysis is sometimes largely compensated for by the excess of weight. Calculation to a dry sample cannot, however, be too strongly advocated for some articles, because it has at all times the advantage of shewing at a glance the harmony that exists between the analyses of samples drawn at different times and containing different percentages of moisture. My close connection with the sugar Associations brings me at times into contact with their officials, and as shewing the good the Associations have already accomplished, I understand the weight of the sugar has improved so much, in comparison with that of former years, that it has even caused them surprise. The improvement is believed to be the result of representations made to the Continental sellers pointing out that the sugar could not have been put into the bags, otherwise it would turn out here. It is obvious that the Continental sellers, now finding that they get value only for the sugar they send, are more careful to make the bags full weight. In conclusion I may add that I have found little or no trouble in connection with any of the analyses which I have executed, and I do not suppose that, with one or two exceptions, there has been any question raised as to the final settlement of value by the results of the analysis obtained. In short, the Beetroot Sugar Association have been so eminently successful in their working that a return to the old *régime* is now quite impossible.

Mr. MACARTHUR said: All the speakers are agreed that the centre of the whole grievance is in sampling. I believe this is perfectly true, and yet Mr. Tatlock, who makes the motion, seems to think that the question of sampling should be left alone, meantime. Now I really cannot understand how—if sampling be such an important matter—it should be left to uneducated men. Would it not be better, therefore, for the committee who are appointed to make representations to manufacturers, merchants and brokers, as well as to chemists, showing that a great deal of the trouble arises through bad samples. I scarcely think that analysts would care to adopt any one method for any particular class of products. I think it is almost suicidal. Circumstances will arise and difficulties will come up in the quality of natural products. For instance, as far as I know just now, there is no common method in print for estimating sulphur in pyrites apart from sulphuric acid. Such a thing is very simple; but there is no recommendation that it should be done; but I think that such a recommendation would fall on the shoulders of this committee, and that their recommendation should be more negative than positive, and should tend to shew the dangers into which an analyst is apt to fall. If apology is necessary for a person speaking on his own special subject, I must apologise for a further reference to pyrites. The committee might recommend a method for the estimation of sulphur, directing that after the pyrites is dissolved the insoluble must be removed by filtration. Now an odd lot might contain a large amount of galena (in which the sulphur of zinc is available); sulphate of lead would be formed; one rigidly faithful chemist would leave this with the useless silica, while another intelligently honest chemist would take care to estimate the sulphur in the insoluble. One would work with rigorous obedience—the other would deviate from strict rule to suit an exceptional case. As to a court of appeal, I scarcely think that would work. Chemists are blamed for being high or low to please their clients; in future, chemists would work to please the court of appeal; therefore, this court would be useless. I cannot agree with what Mr. Milles has stated about precious metals. I think the discrepancies in precious

metals are as great—possibly greater—than in any other class of work; certainly greater than in any other class of work with which I have dealt. It is a very difficult matter indeed to get a fair sample of auriferous or argentiferous material. I remember one case of a product containing a large amount of silver, in which there was the extreme difference of about 500 ounces to the ton between the assays of three very eminent firms. It was certainly a *very* rich sample. Another case: I received a sample of auriferous pyrites from a friend to examine. The sample was rather small, but I got the results to agree to within a few pennyweights (dwts). In sending out the result I stated to him that I hoped that it was close enough for the purpose. He replied that it was perfectly close enough, and that he hoped for my own good I would always get them as bad! It was so much better than he was used to at the gold mine. In conclusion, I would like to say that as sampling is such an important part of the scientific work, it ought to come under the cognisance of this committee, and suggestions and recommendations be made about it.

Mr. STANFORD said: I think the important subject of sampling will necessarily form part of the deliberations of the committee. It has already been brought before the Chemical Section of the London Chamber of Commerce, and it is certainly one of the principal parts of the subject. I was particularly struck with Professor Mills' suggestion about this committee getting chemists to give them some details of their errors. There is no doubt that that is a most important suggestion, and would give them very valuable information. Unfortunately it is not quite easy to elicit it. I remember in the British Association Committee we could not get some chemists to admit that it was possible for them to make an error, and Mr. Tatlock, in his reply, will perhaps tell you a little more about this very important subject of chloride of potassium. He has here shewn you the analysis of chloride of potassium, but I am sure he will bear me out when I say that if he had put on the board the two analyses of a mixed salt containing a smaller proportion of potash, say equal parts of potash and soda, the difference between him and the German chemist would have been very much larger, and this connection brings in the difficulty Mr. Mactear mentioned about manufacturers. Unless this committee is strong enough to carry the manufacturers with them very little will be done. In this particular question of potash analysis I had considered that the British Association Committee thoroughly proved what was the proper method of estimating potash salts. It is a method which I think all British chemists adopt or approve of, and we hear, as a result, that the German manufacturers, finding that this method does not suit them, have combined together and won't have it.

Mr. TATLOCK, replying to the previous speakers, said: At this late hour I shall not detain you long. I was much struck with the remarks of the first speaker (Mr. Mactear) in regard to his experience in the estimation of nitrous compounds by means of the galvanic process which Mr. A. Vernon Harcourt recommended long since, and I am very glad to know that he has got such good results with it, as I have very old experience of it: not in connection with the testing of nitrous compounds in vitriol chamber gases, but in connection with the estimation of nitric acid in saltpetre and gunpowder materials. I had occasion twenty years ago to make hundreds of estimations of nitric acid, existing both in great and in small quantities in saltpetre salts, and I can say that I found that process most invaluable. We had not then the Crum-Frankland methods that we have now, and which

is so useful to most chemists, and at that time, failing it, we adopted the Harcourt process with great success. We are indebted to Professor Mills for the suggestion he has made with regard to recognised figures for permissible errors. In every case, of course, they will vary; but it is very desirable indeed that the justifiable maximum of error should be laid down, if it were for no other purpose than to get people to believe they had got a really good analysis where such had been made. Dr. Mills believes that a perfectly representative sample of a cargo of iron ore cannot be had in the vessel. I can quite sympathise with that as far as my experience goes. Having had to sample about 250,000 tons of iron ore during the last two years I have had some acquaintance with that kind of work, and I have often declined responsibility for the accuracy of such samples. Mr. Miller has stated his experience, which seems to me a very extraordinary one, with regard to the absence of difficulties in sampling ores containing precious metals. I think, however, he has rather unfairly contrasted the discrepancies in the estimations of potassium per cent., such as I have noted here, with those in the determinations of percentages of gold and silver. As I have already stated, these would depend on the particular ingredients determined, and in all cases discrepancies must be compared upon one hundred parts of the special ingredient in question, so that while a difference of a few tenths per cent. might be admissible in a muriate of potash containing upwards of 90 per cent. of chloride of potassium, a hundredth part of that difference would be quite unpardonable in a gold ore containing less than one tenth of a per cent. of the precious metal. It is only within the last week that I made determinations of the gold and silver in two samples of pyrites ore, the amount of gold in both cases being exceedingly small, in fact amounting to only '0001 per cent. It is a very easy matter to determine gold and silver to a very small percentage of the original ore, and there is no excuse, I should say, for discrepancy in the determination of gold or silver beyond very narrow limits on that basis. It is a more difficult matter when we come to some other constituents in samples which we are frequently called upon to analyse, and one of these is the very example he (Mr. Miller) quoted—chloride of potassium. It is quite true there is a difference of 1.4, but this difference, although not permissible, is upon 100 parts, or nearly so, of the ingredients estimated. Had five or six grains, more or less, of gold been returned per ton of ore, it was quite possible that when these ores were wrought on the large scales this difference would have been brought out, and I should be called upon to account for the discrepancy between my analytical and my client's working results. Mr. W. Chrystal suggests that samples should be dried before they are analysed. I perfectly sympathise with that view, and as far as minerals, such as iron ores, are concerned, this has been our practice constantly. However, I did not think it necessary, until quite recently, to dry samples of such articles as commercial muriate of potash before analysis until, indeed, Mr. Chrystal brought it before my notice recently, and I have had occasion to see that he is perfectly correct. The next speaker, Mr. MacArthur, I think, is not to suppose for a single moment that I don't think it very desirable that the International Association should discuss the question of sampling at all. The difficulties that lie in the path are not so much in the way of sampling as in describing a method by which it can be done correctly under all circumstances. I don't think I have anything further to say, but simply to make good an omission in my first re-



marks, to the effect that we must not overlook what others have done in bringing about concordant results and uniform methods in analysis. Mr. Jamieson, who is chemist to the Sussex Agricultural Association, with regard to phosphates, has done much in this direction, and the Society of Public Analysts, who analyse about 15,000 samples yearly, carry on the work with very few discrepancies I must say, and we must not forget that these individuals and bodies have already done very much towards a settlement of this very important question.

The motion, having been put to the meeting, was carried unanimously.

## Obituary.

ROBERT ANGUS SMITH, LL.D., F.R.S.

*A Vice-President and Member of Council of the Society of Chemical Industry.*

On the 11th of April, the venerable Dumas breathed his last. A month later, Monday, May 12th, at a quarter-past ten, Adolphe Wurtz, of Paris, and at half-past ten, Robert Angus Smith, of Manchester, departed this life. Wurtz and Angus Smith were born in the same year, 1817, and both were former pupils of Liebig. It will be remembered that on May 13th (Sunday evening), 1883, Dr. James Young, of Kelly, the most intimate, and one of the oldest of Dr. Angus Smith's friends, passed away, and during Dr. Smith's last moments he referred to this fact.

Robert Angus Smith was born near Glasgow on the 15th of February, 1817, and received his early education in the Glasgow Grammar School. Subsequently he attended the classes of the College of that city. As he shewed a great liking for classics, and more especially Greek, his family thought he was destined to become a minister of religion; but after due reflection the idea was abandoned in favour of the study of chemical science, for which he had early manifested great aptitude. When twenty-two years of age, Robert Angus Smith proceeded to the University of Giessen, and studied chemistry under Liebig. He took the Ph.D. degree, and then returning to England became an assistant to Dr. Lyon Playfair in 1842, in the Lancashire enquiries, conducted under the Health of Towns Commission.

Dr. Smith now settled in Manchester as a professional chemist, and contributed to the proceedings of the British Association, Chemical Society, and other learned Associations. The Imperial Geological Institute of Vienna made him one of its corresponding members, and in 1844 he was elected a Member of the Literary and Philosophical Society of Manchester, in the welfare of which he manifested to the last the warmest interest, and in the year 1864 he was elected president of that Society. In 1856, twelve years after the death of Dalton, he contributed to the Society a biography of that great philosopher, and in 1857 he was elected a Fellow of the Royal Society. He was a Corresponding Member of the Royal Bavarian Academy, Vice-President of the

Chemical Society, and of the Institute of Chemistry. In 1863, Dr. Angus Smith was appointed Inspector-General of Alkali Works for the United Kingdom, and his interesting annual reports have generally contained the results of important investigations. On the passing of the Rivers Pollution Act, he was appointed, together with Sir Robert Rawlinson, Inspector for England and Scotland. The Report for 1882 contains the result of his experiments and researches on water and drainage. His book on "Disinfectants and Disinfection" was published in 1869, and in the report of the Royal Commission on the Cattle Plague was embodied a memoir of his on disinfection. In 1872, Dr. Smith's most widely-read and highly-prized work on "Air and Rain" appeared, the work being designated by him "the beginnings of a chemical climatology." In 1875 a collection of the essays and researches of the late Professor Thomas Graham, F.R.S., was printed and published for private circulation only, by Dr. Smith and his friend Dr. James Young. To this work Dr. Smith contributed a preface, in which the scientific position of Graham is well shown.

In 1879, a work was issued anonymously, entitled "Loch Etive and the Sons of Uisnach." A review, which appeared in the *Inverness Courier* of October 9th, 1879, thus comments on the work: "The book before us, with an unpromising title, and without the smallest amount of assumption on the part of its unnamed author, discusses a great variety of subjects, ancient and modern. It does so with an extent of precision of learning, scientific and historical, rarely to be met with; and, what is rarer still, treats Highland subjects in the calmest and most impartial manner, entirely free from passion and prejudice. At the same time, it shows a playful though subdued fancy, which sheds a pleasing light over dry antiquarian details; and best of all, it displays a large-hearted, a charitable, and a truly catholic Christian spirit towards the living and the dead, while many observations, the result of matured wisdom, are scattered here and there in the most unobtrusive manner." Dr. Angus Smith was the author of the book, which bears witness to considerable archaeological tastes, the paragraph above given, and written of an unknown man, bearing witness to higher qualities of head and heart. In 1882, the University of Edinburgh conferred the honorary degree of LL.D. upon Dr. Smith, to whom some time previously the LL.D. degree of the University of Glasgow had been presented. A detailed account of Dr. Smith's scientific papers appeared some time ago in the *Biograph and Review*. It has been aptly written of the work of Angus Smith's life, that in it he sought "to make known the influence of inorganic and organic compounds alike upon life, to reconcile industry with health and beauty, to renew the flowers and the greenery of trees, to freshen the rivers, and to restore vigour and cheerfulness to the jaded citizen and tired worker." Dr. Angus Smith was a great authority on the peat, brown coal, and lignite deposits in Europe, and the composition, formation and uses of peat formed subjects of special

interest to him. His essay, entitled "A Study of Peat," contributed to the Memoirs of the Literary and Philosophical Society of Manchester, is an interesting and instructive treatise, abounding with the quaint bright originalities and simplicities of style and idea so characteristic of him, surrounding which is frequently found an atmosphere of quiet humour, perfectly under control, and never allowed to become in the least degree extraneous to the subject treated of. Just in the simplicity and quaintness of his style of writing lies his strength, his sentences being short, trenchant, and uninvolved; in fact, Angus Smith wrote very nearly as he thought and spoke. In the essay on peat, the following sentence occurs, and it fully bears out what has just been said:—"By destroying peat-bogs on hills, we destroy valuable reservoirs made cheaply and maintained cheaply—that is at the price of the rent of the land at most, but not always at this price. If we destroy them, and make reservoirs below the level of the surface, we know something of the danger as well as expense. A peat bog, ten feet deep, will hold as much water as a reservoir of the same dimensions, seven and a half feet deep; it requires no digging, and may be used for shooting over, and for health. It retains the water better in summer than our reservoirs; it is, in fact, a covered reservoir, and would make a valuable covering to those hills which have works below continually demanding fresh streams. One of my proposals, therefore, is to grow our water-reservoirs instead of digging them."

As Chief-Inspector of Alkali Works, it is needless to say a man was required who combined in himself a true, kindly, and conciliatory disposition, with great tact, discretion and sufficient reticence. From what has been already said of Dr. Angus Smith, it will be seen how well fitted he was for the post, especially in the earlier stages of its development.

One of the distinguishing features of Dr. Angus Smith's character, was what may perhaps be termed, an anxious care for the truth in small things as well as in great. He knew that the safety of the great things was ensured, if their lesser details were founded on truth. Being very cautious in giving opinions, as well as very careful of the feelings of others, and having a great reverence for truth as truth, it may be readily imagined that on many topics freely discussed, condemned, or advocated by most people, he could not be persuaded to speak to a definite opinion—he preferred reticence.

Dr. Angus Smith was never married; but in his nephews and nieces he took a paternal interest, and this interest was also extended to the family of his friend Young, by whom his counsel and wisdom are regarded as oracular. He delighted to assist young men, and to encourage and influence them in what he thought was their right course.

His last moments were quite free from pain, and consciousness was preserved to the end. From his window he could see the advance of spring, the clothing of the shrubs and trees with green, and he was much interested in watching two birds building their nest in full view of the window.

Towards the close, when one inquired of him how he felt, he replied in a faint voice, and yet with a tinge of the old quaint humour, "Thinking and sinking."

Perhaps more especially because of his usual reticence on such matters, inquiries have been made concerning his religious views. The recital of a little episode may serve to throw light on this matter. A scientific memoir had been read to him, in the course of which reference was made to the influence which the positive religious beliefs of the person forming the subject of the memoir had exercised upon his success as an investigator. "I am glad you have written that," exclaimed Dr. Smith with enthusiasm; "it brings my mind nearer to the mind of the writer." W. S.

## Journal and Patent Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Chemical Fire Engines.* J. Gibbs and D. Fotheringham, Glasgow. Eng. Pat. 3844, Aug. 7, 1883. Provisional protection granted only.

THIS invention relates to an improved method of working those fire engines termed "extincteurs," which depend for their action upon the pressure of carbonic anhydride expelled, from the alkaline carbonate with which they are charged, by a small quantity of an acid. Hitherto the acid has been suspended in the interior of the vessel in a bottle, which is broken by shaking or canting at the desired moment. In the present case the patentees propose to effect this by providing for the acid a cylindrical vessel, having a valve at its lower extremity, which can be opened by a spindie passing through a stuffing box from the outside, and thus more readily discharge the acid into the alkaline solution. Two forms of hollow plug cocks, containing the acid in their interior and worked in a similar manner, are also described.—C. C. H.

*Apparatus for Grinding or Reducing Ores, Phosphates, Quartz, etc.* J. Wood, West Stockwith, Nottingham. Eng. Pat. 4002, Aug. 17, 1883.

THIS invention consists of a perforated revolving pan, the perforations being conical and the smaller end towards the inside of the pan; a pair of edge runners are placed resting thereon and free to turn on their axes. The runners are made as heavy as possible, and with narrow edges, so that material introduced to be ground being subjected to the concentrated weight of the runners in a very small space, reduction to powder is effected without much friction or attrition. Passing through the perforated bottom of the pan, and falling on the bed of the machine, the ground material is removed by a revolving scraper into an elevator which discharges into a hopper placed some distance above the revolving pan. The hopper communicates at its lower part with suitably constructed settling chambers, which are partially exhausted by a fan, and so arranged that the induced current of air carrying with it the fine particles of the ground material traverses the chambers in a zig-zag fashion. The fine particles so drawn into the chambers gradually deposit, and can be withdrawn from time to time. The heavier portions of the ground material pass out through the bottom of the hopper, and after traversing an inclined guide formed by sloping partitions placed alternately towards the right and left, so as to afford a circuitous path, are delivered to be reground upon the revolving table of the machine. The patentee claims separately—(1) the apparatus consisting of the revolving



table and runners as described; (2) the settling chambers whereby the material is deposited by being subjected to the action of an induced current of air; (3) the combination of the various apparatus as and for the purpose substantially described.—C. C. II.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Note on the Employment of the Abel Petroleum Testing Apparatus in Tropical Climates.* By Sir Frederick Abel, C.B., D.C.L., F.R.S., and Boventon Redwood, F.C.S., F.I.C. Chem. News, May 2, 1884.

ONE of the authors has shewn (see this Journal, vol. I., p. 473—478) that the method of testing prescribed by the Petroleum Acts of this country and India is liable to furnish, with one and the same sample of oil, lower results in a tropical country than in a temperate climate, unless certain precautions not specified in the legal directions for applying the test are observed. It was found that a sample of petroleum which gave a flashing-point of 73° F. when the test was made in the cool atmosphere of the laboratory, flashed on the first application of the test flame at 66° F. when the testing was conducted in an apartment heated to a tropical temperature. It was found that when the sample was subjected to prolonged cooling prior to testing, the tendency to exhibit a lower flashing-point was largely diminished. A modification suggested by Dr. Lyon to avoid a false flash consisted in leaving the testing slide withdrawn for some time before commencing the application of the test-flame, and when experimenting under these conditions it was found that the depression of the flashing-point was no longer distinctly shown. It was therefore clear that the lower flashing-point was due, at any rate very largely, to vapour disengaged prior to the first application of the test-flame, and that this vapour became dissipated when the modification referred to was adopted.

With the continued assistance of Dr. Lyon the relative merits of various methods of overcoming this effect of a tropical climate upon the flashing-point were ascertained. In addition to the plan already described, the following methods were found available:

(1) The removal of the collected vapour by means of an aspirator after placing the cover on the oil-cup.

(2) The dispersal of the vapour by gently blowing over the surface of the oil after filling the cup and before putting on the cover.

(3) The commencing of the application of the test-flame at a temperature considerably lower than that prescribed by the Acts. The plan of blowing away the vapour was found simple and effectual in careful hands, but it was not thought a desirable one to adopt, being obviously liable to abuse, and, with Dr. Lyon, the authors were ultimately led to give the preference to the method of commencing the application of the test-flame at a temperature of 56° F., when the first experiment has furnished a flashing-point below 73° F. The effect of this modification, as well as that of any one of the alternatives above mentioned, is to gradually remove the vapour disengaged in the filling of the cup in successive quantities too small to give a flash, each application of the test-flame determining a current of air through the upper part of the cup, and sweeping out a portion of the gaseous contents. Any one of the modes of operating above described is obviously open to the objection that it effects the removal of some small proportion of the most volatile constituents of the oil (which in all instances is a mixture of liquids differing, and in some cases very considerably, in volatility), and that consequently the character of the sample has sustained some alteration, tending to raise its flashing-point before the application of the test-flame is actually commenced. This is, however, unavoidable, and must even occur in tropical climates, to some extent, whenever a sample is subject to any kind of manipulation in the open air. The extent to which the flashing-point is

thereby effected is, however, unimportant in comparison with the value to be attached, from a commercial point of view, to the attainment of uniform results in the examination of cargoes of oils in different countries. The method of operating just given will yield results fairly concordant, even when operators employing it are under different circumstances of climate; but still the authors felt bound to point out that the Abel test could not give precisely concordant results under such circumstances, the flashing-points in a tropical country being somewhat lower than those found in the temperate zones. It was recommended, therefore, that the trade should provide for the liability of some samples to exhibit, when tested in India, a flashing-point as much as three degrees lower than the flashing-point recorded before shipment. It is pointed out that the amount of vapour disengaged in the filling of the cup obviously depends upon the precise mode of manipulation, and is, as might be expected, greatest when the oil is poured in slowly in a thin stream from a vessel held some inches above the cup. Under these conditions the considerable agitation to which the liquid is subjected acts mechanically in promoting the disengagement of vapour, while at the same time the attenuated volume of the liquid is exposed to a temperature much above the flashing-point of the oil. These circumstances may combine to cause a layer of vapour to cover the surface of the oil in the cup before commencement of the test.

W. S.

*The Determination of Phenol in Creosote.* Kleinert. Zeit. Analyt. Chem. 23, 1-13.

WHILST examining a number of creosote oils, the author found that the determinations of the acid oils and of phenol by Koppeschar's method by no means gave concordant results, as is shewn by the following percentages:—

Acid Oils	18	30	18	48	22	22	22	17	24	20
Phenol ..	13.7	28	16	50	16.98	17.25	13	18.45	12.75	17.17

The acid oils collected during these experiments should contain almost exactly 75 per cent. of phenol, and accordingly the greater bulk should have dissolved when a portion was shaken with twenty times its volume of warm water. This, however, was not the case: the greater portion remained undissolved, and this when heated began to distil over at from 208° to 215°. These observations show that the acid oils insoluble in water contain a body boiling at a considerably higher temperature than phenol, but acting as phenol does toward bromine. After describing in detail a number of fractional distillations and the examination of the fractions, the author sums up: (1) The very small portion, viz., 1 to 2 per cent., distilling over between 150° and 200° shows that these creosote oils contain but very little phenol. (2) It cannot be supposed that the distillate coming over above 200° contains phenol (whose boiling point is 184°) in anything more than small quantity, and yet not only is the fraction boiling 200° to 250° the largest, but it is also the one which shows the highest test for phenol by the bromine method. (3) The acid oils boiling above 250° also contain substances behaving exactly like phenol towards bromine. Consequently when phenol is determined in creosote oil according to Koppeschar's method, the number obtained is very considerably too high, owing to the presence of bodies of higher boiling point than that of phenol, precipitated by bromine, and soluble in caustic soda, but insoluble in water.—H. B.

*The Occurrence of Pyridine in Commercial Ammonia.* H. Ost. Zeit. Analyt. Chem. 23, 59-60.

IN order to detect this impurity the ammonia is nearly neutralised, when the pyridine smell can be noticed. By distilling the liquid, collecting the distillate in hydrochloric acid, evaporating, and extracting the residue with absolute alcohol, a solution is obtained containing but little ammonium chloride. This last is removed by addition of platinum chloride, and on evaporation of the alcoholic filtrate the platinum-pyridine double chloride separates in characteristic forms.—H. B.

## IV.—COLOURING MATTERS AND DYES.

*The Assay of Pure Indigo Samples by Means of the Spectroscope.* C. H. Wolff. *Zeit. Anal. Chem.* 23, 29-32.

THE author has repeated with some alterations his previous experiments (*Zeit. Anal. Chem.* 17, 310). The slit of the instrument was constructed after Krüss's method, and the intensity of the light measured by Vierordt's method. A prism of less dispersive power and a slightly different portion of the spectrum were employed. The indigo samples previously employed were re-examined and compared with a sample of artificial indigo, crystallised from aniline by Baeyer, which served as the standard; the sample previously employed as standard was No. 2:—

	Per cent.
Indigotin, Baeyer .....	100.00
Indigotin, Trommsdorff .....	91.58
Indigotin, sublimed, Schuchardt .....	83.41
Indigotin, sublimed, Wolff .....	82.45
Indigotin, Vierordt .....	81.95
Indigotin, crystallised, Schuchardt .....	79.08

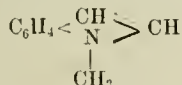
H. B.

*Synthesis of Quinoline Derivatives.* L. Kuorr. *Berl. Ber.* 17, 540.

IN reference to a new synthesis of quinoline derivatives by the action of aniline of aceto-acetic ether recently announced by the author (*Berl. Ber.* 16, 2593), he now publishes the results of further experiments. From these it appears that the same reaction takes place between most primary aromatic amines and carboketonic acid ethers of the general formula  $R_1-CO-CH(R_2)-COOR_3$ . The reactions between aniline, orthotoluidine, paratoluidine, betanaphthylamine, and orthophenylenediamine respectively, with aceto-acetic ether, and between paratoluidine and succino-succinic ether are described in the paper.—A. S.

*Synthesis of Indol Derivatives,* Emil Fischer and Otto Hess. *Berl. Ber.* 17, 559.

IN a previous paper the authors have described the formation of an acid by the action of methylphenylhydrazine on pyrrolic acid, which by the action of hydrochloric acid splits up into ammonia and a new acid possessing the formula  $C_{10}H_9NO_2$ . Further investigation has shown this latter compound to be a derivative of indol. By continued heating it loses  $CO_2$ , and yields a weak basic substance,  $C_9H_7N$ , very closely resembling indol. This on oxidation gives methylpseudoisatin, and from this it is inferred that the base is methylindol—



and the original acid methylindolecarboxylic acid. The formation of indol derivatives from pyrrolic acid and secondary aromatic hydrazines appears to be a general one, and one likely to be of importance for synthesis of bodies belonging to the indigo group. The preparation and properties of the above-named compounds, and other similar ones are described in detail.—A. S.

*Improvements in the Production of Certain Substances for Use in Obtaining Colouring Matters suitable for Dyeing and Printing.* Robert Holliday and W. R. Hodgkinson. *Eng. Pat.* 3730, July 31, 1883.

THE inventors claim the production of the phenols of fluorene and acenaphthene by converting these hydrocarbons into monosulphonic acids and fusing with alkali. They claim also the amido derivatives of fluorene and acenaphthene (fluorylamines) obtained by nitrating and reducing the nitro derivatives of these hydrocarbons. The fluorols and fluorylamines or their sulphonic acids are employed for the production of azo colours in the same manner as the naphthols and naphthylamines.

R. M.

*Improvements in the Preparation of Colouring Matters suitable for Dyeing and Printing.* Astley Paston Price, Lincoln's Inn Fields. Communicated by Heinrich Caro, Mannheim, Germany. *Eng. Pat.* 4428, Sept. 15, 1883.

By acting upon dimethylaniline, diethylaniline, or other tertiary monamines with phosgene gas,  $COCl_2$ , in the presence of anhydrous aluminium chloride colouring matters are produced. Thus, in the case of dimethylaniline 20 parts of the latter base are mixed with 5 parts of aluminium chloride, the temperature not being allowed to rise above  $30^\circ C$ , and 3 parts of carbon oxychloride gradually added. The temperature is kept at  $20^\circ$  to  $30^\circ C$ . for five or six hours, by which time the reaction is complete. The colour is purified by the usual methods, and then forms a violet dye which is said to differ from the ordinary "methyl-violets" in its bluer and more brilliant shade, and in its greater power of crystallisation.—R. M.

*A New or Improved Manufacture of Yellow and Orange Colouring Matters.* John Imray, Southampton Buildings, Chancery Lane. A communication from "La Société Anonyme des Matières Colorantes et Produits Chimiques," of St. Denis and Messrs. L. Roussin and A. Rosenstiehl, of Paris. *Eng. Pat.* 4621, Sept. 28, 1883.

THIS invention relates to the production of soluble azo dyes containing the  $COOH$  group instead of the  $HSO_3$  group. As an example the inventors give the production of colouring matters from para or meta-nitrobenzoic acid, which is diazotised and combined with diphenylamine or benzyaniline in the usual way.—R. M.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Manufacture of Chlorate of Potash.* E. K. Muspratt and G. Eschellmann. *Eng. Pat.* 3960, Aug. 15, 1883.

MAGNESIA suspended in water is substituted for milk of lime ordinarily employed. Chlorine is passed into the mixture, and the resulting liquor, which contains magnesium chlorate and chloride, is treated with potassium chloride. The greater portion of the chlorate of potash so produced is separated by crystallisation, whilst the rest is deposited on evaporating the mother-liquor. The liquor, which after removal of the chlorate of potash contains only magnesium chloride, is allowed to solidify, and is then reconverted into magnesia by heating in the known method. The magnesia can thus be used repeatedly, a slight contamination with magnesium chloride not affecting its utility.—A. S.

*Manufacture of Chlorine.* W. Weldon. *Eng. Pat.* 4079, 1883.

THE solution of manganese chloride obtained in the preparation of chlorine by the action of hydrochloric acid on oxide of manganese is freed from excess of acid by digestion with regenerated oxide, or by passing down a tower in which hot gases are ascending. The solution is then evaporated to dryness, and the residual product should not contain more than one equivalent of water. It is then reduced to powder, and either alone or mixed with powder of oxide of manganese resulting from the next operation, is heated (most conveniently in rotating cylinders) in a current of air. Chlorine is thereby evolved, and there results an oxide of manganese or mixture of oxides which may be used for the fresh generation of chlorine.—A. S.

## X.—METALLURGY, MINING, Etc.

*A Method of Distinguishing between Iron and Steel in Small Pieces.* *Dingl. Polyt. Journ.* 251, 332.

GENERALLY the appearance of a fresh fracture serves for the classification of sample pieces, but this test is not



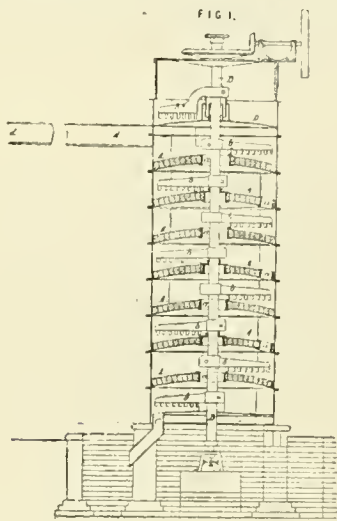
sufficiently accurate when it is a question of good fine-grained iron or very soft steel. To make the discrimination in such cases easy and sure, Walrand proposes to heat the sample until, on cooling, it assumes a blue colour, and then observe the fracture. By this process all doubt as to the nature of the piece of metal in question is removed. The experiment can be made in the following way: The sample rod, about 25cm. to 30cm. long, is slightly filed about 4cm. to 5cm. from its ends; the one end is then heated slowly and uniformly to a dark red heat ( $325^{\circ}$  to  $400^{\circ}$ ) and cooled in water. While cooling, the still warm piece of metal must be repeatedly tested with a file until the clean surface appears dark yellow, or better, blue, when it is quickly and completely cooled. The surfaces exposed by breaking off the ends of the rod at the file marks can now be compared. Ordinary cold broken wrought iron appears fibrous or grainy, but when it has been treated in the above manner the fracture looks dull, torn and short, fibred. Hard and moderately hard steel is fine grained; after heating and tempering it has a glancing, wholly, or partially, smooth fracture. Swedish iron has only a trace of fibre and consequently cannot be distinguished from soft steel, when tempered however the fibres can be distinctly seen and the smooth appearance vanishes, while with soft steel, similarly treated, this appearance becomes more distinct.—W. M.

*Cast Steel.* Dingl. Polyt. Jour. 250, 283.

MESSRS. GLÖCKNER, of Tschirndorf, prepare a metal, by fusing together cast iron and steel, which is well adapted to the manufacture of agricultural implements. Tested for breaking strain under the usual conditions, it gave the following results: The metal to be tested was cast in rectangular bars, of 31mm. square section, which were found to break under the several strains: (1) bending, at 33.1 to 39.9 kilos., per 1 square mm.; (2) drawing, at 22.3 to 23.5 kilos.; (3) compressing, at 107.8 to 110.1 kilos. The limit of elasticity was reached under a load of 4.6 to 4.96 kilos. per 1 square mm.—C. F. C.

*Furnaces or Apparatus for Burning, Calcining, or Roasting Sulphur Ores, Spent Oxide of Iron, etc.* J. S. McDougall, Manchester. Eng. Pat. 3985, Aug. 16, 1883.

THE subject of this invention is the construction of furnaces for burning or calcining ores, pyrites, or spent oxide of iron from the purification of coal gas.



The first modification described consists of a number of superposed floors having a central shaft passing vertically through them, provided with horizontal

rakes furnished on their faces with inclined teeth, and driven by any form of gearing from outside the furnace. The floors communicate alternately with each other by a central opening in the one floor and a circumferential opening in the next floor, and the teeth of the rakes are alternately so inclined that the material passing through the furnace is on the one drawn towards the centre, and in the other pushed outwards towards the circumference. Circulation from one floor to the other is thus established throughout the furnace, so that material entering by the hopper at the top after travelling over the whole surface of each floor, is discharged at the bottom. Dust chambers provided with perforated baffle plates for the interception of dust carried over by the draught are also described, with arrangements for drawing out the settled dust so as to prevent egress of gas or admission of air. The shaft and rake-arms are constructed of cast-iron, having a central wrought-iron tube in order to obviate warping or bending from the effect of heat, and in order that the shaft may be readily withdrawn, the arms or rakes are fixed thereto by a forked-shaped end and cotter. A second modification is described for such materials as small pyrites or spent oxide so as to avoid the dust occasioned by the vertical passage from floor to floor. The furnace is constructed of an oblong horizontal floor or chamber, provided with a series of vertical shafts, having rakes similar to those before described, and revolving in opposite directions. The teeth are so placed as to draw the material towards each shaft, and thus pass it from one to the other, and from end to end of the furnace. Where prolonged roasting or burning is requisite, a similar furnace, or the first modification, may be superposed above the last-mentioned through which the material is first passed. The points of novelty claimed are: (1) The construction of the form of furnace with superposed floors provided with agitators as described. (2) The lining of the shafts and rakes with a wrought iron tube. (3) The mode of attachment of the rakes or blades. (4) The dust-separating chambers. (5) The combination of either modification with the dust chambers as described. (6) The form of furnace having a horizontal chamber as described either separate or in combination with a similar form of furnace superposed thereon.—C. C. H.

*Ore Roasting Furnaces.* H. H. Lake, London. (Communicated by T. Walker and J. F. Carter, Philadelphia, U.S.A., Eng. Pat. 4000. August 17, 1883.)

THE object of this invention is to effect in a rapid, automatic, continuous, and economical manner the roasting, oxidation, desulphurisation, etc., of ores, or the generation of sulphurous acid from pyrites. This is carried into effect in the following manner: Horizontal retorts are placed one above the other over a furnace, the ends being built into the brickwork, and sufficient space being left between each for the proper circulation of the heated gases from the furnace. The spaces round the retorts communicate with each other through openings made at alternate ends of the retorts, so that the furnace gases traverse the spaces from end to end in a circuitous path on their passage from the furnace to the flue. Two or more such series of retorts may be used. Hollow shafts, carrying rakes having inclined teeth, pass through the centre of each retort, and these are secured in suitable bearings at each end of the retort, and are rotated by means of a worm and worm wheel on one of their ends outside the furnace. The alternate ends are provided with connection pipes so that during the operation of the furnace a current of water may circulate through the hollow shafts which are thereby kept cool and prevented from warping. Each of the retorts communicates with a dust settling chamber so that the finer particles which may be carried away by the current of air found necessary for the operation, and introduced at the ends of each retort, may be recovered. Each of the retorts communicates with the other at alternate ends by a pipe, so that the material introduced into the top retort traverses its whole length before it is discharged into the next, along which it is forced in the opposite direction

and escapes into the next lower retort at the opposite end to that into which it is introduced. The working of the apparatus is as follows: the broken ore being introduced into the top retort from a hopper is, by the revolution of the hollow shaft and rake described, carried along

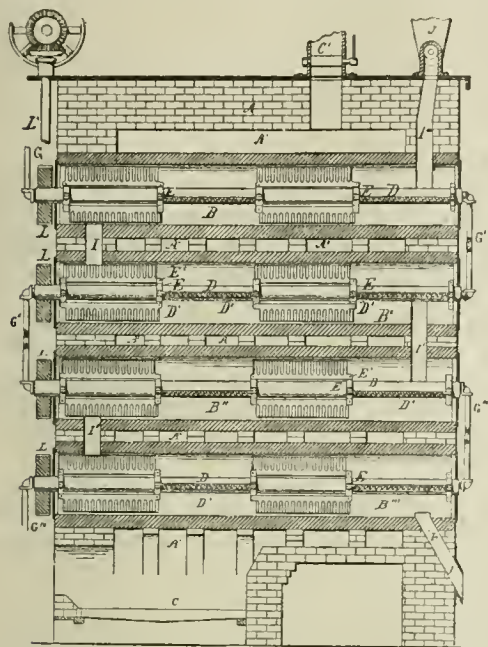


Fig. 1.

to the opposite end; having arrived here it drops into the next retort, and is again similarly carried along its entire length, escaping through the vertical passage into the next lower retort, and so on through the whole series until finally discharged from the last of the series. In order to prevent displacement of the hollow

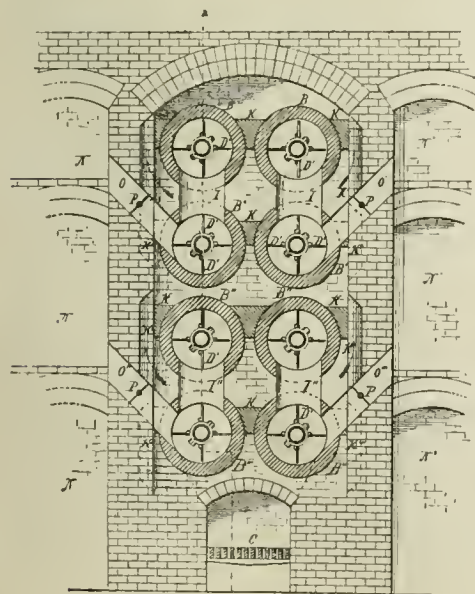


Fig. 2.

shafts and rakes by expansion, the connecting water pipes are provided with a bend of sufficient curvature to allow for all possible expansion. The motion

of the rakes not only produces an advancing motion of the ore, etc., but also continually exposes a fresh surface to the heated air of the retort. The invention claimed is: (1) The combination of the retort, hollow shaft, rakes and dust chambers as described, so as to work in a continuous manner. (2) A series of such retorts, etc., connected with each other and provided with suitable air openings at the ends of the retorts. (3) The connection of such a series of retorts with the dust chambers and apparatus to regulate the draught thereinto from the retorts. (4) The combination of the retort, hollow shaft and rakes, together with the mode of fastening the latter to the shaft as described.—C. C. H.

*Crucible Furnaces, etc.* Provisional protection granted only. B. J. B. Mills, London. Communicated by G. Fischer, Hainfeld, Austria. Eng. Pat. 3585, July 21, 1883.

A HEATING stove is provided, consisting of hollow cylinders of refractory material forming a part of the furnace in which the charge is previously heated, so that the bursting of crucibles after their introduction into the furnace is prevented; a more regular working of the furnace is also obtained. Improved methods for regulating the blast, consisting of a perforated plate beneath the fire-grate, arranged so as to turn from the outside, are also described. Peculiarly-shaped vessels, called trans-fusion vessels, are also provided, into which the melted charge is poured after the operation is finished, and these being previously heated, the metal can be conveyed from the furnace without loss of heat.—C. C. H.

*Amalgamating Process for the Extraction of Gold and Silver from their Ores and Apparatus Employed therein.* John Needham Longden, William Pritchard Morgan, and Archibald William Stirling, Charter's Towers, Queensland, and London. Eng. Pat. 3986, Aug. 16, 1883. This invention received provisional protection only.

IN the processes hitherto in use it has been found that the ascent of ore through the mercury of the amalgamating pan is so rapid that the full amalgamating effect of the mercury cannot be obtained. To obviate this, there is introduced into the pan and immersed in the mercury above the level at which the ore is introduced (that is to say, at which the ore is discharged into the mercury) an amalgamating coil or coils, or bars, or gratings, or suitably-curved plating made of copper or electro-silver conveniently arranged and of suitable shape, by which means we retard the ascent of the ore through the mercury and regulate its speed and direction.—J. T.

*Improvements in the Preparation of Selenium for, and the Manufacture of, Selenium Cells for Electrical Purposes, and Apparatus therefor.* William Robert Lake. A communication from Charles Edgar Fritts, and Daniel H. Hopkinson, New York, U. S. A. Eng. Pat. 3249, Feb. 13, 1884.

THE selenium, which should be as pure as possible, is formed into thin sheets of any desired form by inclosing it between blocks or plates of the desired form having smooth polished surfaces, applying heat till it softens, pressing the plates together to bring the selenium into the required shape, continuing the heat until the second melting point is reached, again applying pressure to reduce the selenium to the required thickness, and then allowing the whole to cool slowly. To prevent cracks in thin sheets of selenium on removing from the pressing plates, thin plates of mica may be placed on each side of the selenium, or thin sheets of metal may be used which can be readily removed by a solvent therefor which will not affect the selenium. The selenium is sometimes pressed and formed for use on a permanent metal back plate. The apparatus for heating the selenium consists of a muffle, the lower portion of which is charged with scrap iron to equalise and retain the heat, a stirrup with



a screw projecting through the top of the muffle, and a thermometer. The plates containing the selenium are placed in the stirrup and pressure is applied by the screw when the proper temperature has been reached. Examples of selenium cells are given in some, of which the electric current is brought up to the selenium by means of a conducting fluid, and is taken away similarly. In other cells the thin plates of annealed selenium can be used in what may be termed dry cells, by coating them on one or both sides with a metal capable of being made sufficiently thin to permit light rays to pass through it to the selenium. Platinum, gold, or silver is suitable for this purpose, to be applied to both sides of thin sheets of selenium, or to the surface of the selenium when it is attached to a metallic plate.—J. T.

*Process and Apparatus for Separating and Treating Metals and Extracting them from Ores, Mats, etc.*

Henry Renner Cassel, New York, U.S.A. Eng. Pat. 3873, Aug. 9, 1883.

THIS invention proposes to treat gold and other ores with a liquid which under electrolytic decomposition yields chlorine in any apparatus in which the anodes and cathodes are separated by any porous material capable of acting as a filter, or in which the anode and cathode compartments consist of separate vessels suitably connected, the ore to be treated being always placed in the anode compartment and kept in motion either by stirrers preferably made of graphite or carbon, or by making the anode compartment revolve, or the ore is otherwise kept in motion for the purpose of enabling the chlorine to reach every particle of the metals contained in the ores or other substances to be treated. The apparatus is used in connection with a dynamo-machine or other source of electricity, such apparatus being made either with the object of dissolving the metal and keeping it in solution, or with the object of depositing the dissolved metal at the negative pole. Amongst other things the inventors claim the process for treating metals, ores, and auriferous compounds, which consist in dissolving the metals in a solution yielding chlorine under electrolytic decomposition, and in preventing the dissolved metals reaching the negative pole by interposing between the electrodes a porous material as described. Drawings of proposed apparatus are given.—J. T.

*Treatment or Reduction of Iron Ores for Obtaining Iron or Steel therefrom.* William Arthur, Cowes. Communicated from Joseph Pearson Gill, New York, U.S.A. Eng. Pat. 4140, Aug. 28, 1883.

THIS invention consists essentially in the application in measured and automatically regulated quantities of hydrogen, nitrogen, carbonic oxide, and carbonic acid gases and air with hydrocarbon vapours, and either with or without steam; to iron ores of any kind or description, in closed muffles, retorts or chambers from which the outside air and the products of combustion are excluded; and also in the treatment of such ores in open hearths in cupolas and in reverberatory furnaces for the separate steps required in the treatment of the respective ores and metals.—J. T.

*Compound Metal or Alloy, chiefly Designed for Deoxidising and Coating Metal Plates.* Henry Harris Lake. Communicated by John Benlow Jones, Brooklyn, N. Y. U.S.A. Eng. Pat. 4057, Aug. 21, 1883. (This invention received provisional protection only.)

THE inventor makes a bath of mixtures of metallic sodium, tin, lead and zinc in varying proportions, and immerses therein the articles, of iron mainly, to be coated. Owing to the low melting point of the bath, a covering of oil or fat can be applied to the bath to prevent oxidation.

J. T.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*Detection of Hydrocarbons in Fats.* Dingl. Polyt. Jour. 251, 335.

FOR the estimation of mineral oils, paraffin, etc., in fats, F. Nitsche heats 10grms. of the fat under examination, with 7grms. of soda solution 38° B., and 30grms. 90-96 per cent. alcohol in a water-bath until the alcohol begins to boil, and he then adds slowly 40grms. glycerine, 28° B., 10 c.c. petroleum spirit is added to the soap solution, which appears turbid, if there be an appreciable quantity of hydrocarbons present, and the mixture is thoroughly shaken. The petroleum spirit dissolves the hydrocarbons and separates easily and completely from the soap solution which does not solidify at ordinary temperatures owing to the addition of glycerine. On evaporating the petroleum spirit, the mineral oil remains behind. For quantitative analysis it is more exact to saponify 10grms. of the fat, separate the fatty acids, titrate and compare the quantity of alkali used, with that required by the glycerine soap, which has been washed with benzine, and decomposed by boiling with sulphuric acid. This process can also be used in testing stearin and other candles for paraffin, ceresin, mineral wax, etc., and has the advantage that the residue, after driving off the petroleum spirit, is left unchanged, and its physical and chemical properties can be examined.

W. M.

## XIII.—TANNING, LEATHER, GLUE, AND SIZE.

*An Improved Process of Tanning.* A. M. and W. Clark. A communication from Gandenzio dalla Zonca, Venice. Eng. Pat. 3829, Aug. 4, 1883.

THE hides and skins, after being fleshed and depilated, are first coloured by immersion in weak liquor which has served in previous tanning operations, to which is added about five ounces of ground vegetable substances containing tannin marking 4° or 5° Baumé. This operation requires only eight or nine days instead of several months, as in ordinary. The tanning operation is thus performed: The hides or skins are first sewn together by their opposite edges with hemp, or so as to form a kind of bag, leaving an opening at the hind legs only, at which is introduced a mixture of ground vegetable substances containing tannin. The hides are then laid on their backs in groups, for the sake of convenience, with all the heads in a line, in large sheds sheltered from strong air currents, care being taken to prevent their becoming wetted on the outside. Liquor from previous tanning operations is then introduced through connections adapted to the openings in the legs, by means of pipes. The liquor is heated to 25° to 30° R. in spring and summer, and to from 30° to 35° R. in autumn and winter. The openings in the hind legs are closed by suitable means, after the skins have been filled about one-third full of liquor. The skins are then oscillated either by hand or mechanical means for some minutes, in order to mix the liquor with the tanning materials previously introduced. After remaining an hour fresh liquor is introduced until they are completely filled, and the oscillation is repeated. An hour and a half afterwards a fresh quantity of liquor is introduced, so as to distend the skins, after which the openings in legs are closed. These filling and shaking operations are repeated every two hours, in order to compensate for the liquor lost by drainage, and also to keep the liquor at a constant temperature. The above operations are continued for from three to six days, according to purpose for which the leather is required. By this invention skins used for uppers may be completely tanned in 15 to 16 days, and hides for sole leather, etc., in from 24 to 26 days.—A. W.

## XIV.—AGRICULTURE, MANURES, Etc.

*Lime and Magnesia in Plants.* E. v. Raumer. Centralblatt f. Agrikultur chemie. 13, 46-47.

ACCORDING to the author's observations on the seedlings of *Phaseolus multiflorus* the lime is concerned in the

building up of new material required for growth and the strengthening of the cell walls. The formation and change of starch is independent of the presence of lime; but magnesia plays a part in the formation of starch in, and the removal of starch from the chlorophyll grains, and in the formation of the same. This is shown by the plants becoming pale in colour, and the formation of chlorophyll ceasing if magnesia be not supplied, and further Hoppe-Seyler has found magnesia to be a constant constituent of chlorophyllan.—H. B.

*On Phenylamidopropionic Acid, Amidovalerianic Acid, and several other Nitrogenous Constituents of the Seedlings of Lupinus luteus.* E. Schulze and J. Barbieri. Central-blatt f. Agrikultur chemie 13, 65.

BESIDES asparagin which has long been known to occur in these seedlings, the author has shewn the presence of the two above-mentioned acids, they occur principally in the axis, in the cotyledons is a body apparently leucin but in very small quantity. Bodies of the xanthin group occur in both axis and cotyledons. Lecithin occurs in the axis, and probably also in the cotyledons. Peptones occur in various parts, but only in minute quantity.

H. B.

*Method of Treating Calcareous Phosphorites or Ores for converting the Carbonate of Lime contained in the same into Phosphate of Lime.* William Robt. Lake, London. Eng. Pat. 5363. November 13, 1883.

THE desiccated and finely pulverised ore is treated with sufficient phosphoric acid to convert the carbonate into soluble phosphate of lime. The mixture may (a) be treated with water and filtered, thus yielding a solution of soluble phosphate and a solid residue of tribasic phosphate, or (b) it may be treated with sulphuric acid for the conversion of all the phosphoric acid into soluble phosphate or free phosphoric acid, or (c) it may, after the addition of sulphuric acid, be mixed with water and filtered to separate the sulphate of lime and other insoluble matters from the phosphoric acid and soluble phosphate.—A. R. D.

## XV.—SUGAR, GUMS, STARCHES, Etc.

*The Manufacture of Maize-starch as a New Agricultural Industry.* L. von Wagner. Dingl. Polyt. Journ. 250, 173.

THE direct object of an agricultural industry is the production of food, the indirect, the obtaining of manure. Nitrogenous matters are the most important, both for food and for manure, and the author contends that the manufacture of maize-starch satisfies both these requirements. In this manufacture, the 8 to 15 per cent. of nitrogenous matter contained in the maize is obtained as a bye-product in a fresh and unchanged condition. This, when mixed with chopped straw, maize-roots, husks, and shoots, makes an excellent and easily digestible food for cattle. Maize yields 52 to 60 per cent. of starch as a main product; and, as a bye-product, 8 to 15 per cent. of nitrogenous matters; and, as residue in husks and shoots, 12 to 18 per cent. At the present time agricultural starch manufactories obtain their product from potatoes. A comparison drawn between the potato-starch and maize-starch industries is very significant. Good potatoes yield 25 per cent. of their weight of dry substance, and contain 1.1 per cent. of nitrogenous matter. Therefore, 100 kilogrammes of dry substance gives 4.4 kilogrammes of nitrogenous matter. Maize yields 77.6 per cent. of dry substance, and contains 10.6 per cent. of nitrogenous matter, so that 100 kilogrammes of the dry substance gives 13.6 of nitrogenous matter, or three times as much as potatoes. The potato bye-product yields 11 per cent. of dry substance, and 0.5 per cent. of nitrogenous matter, so that the dry substance contains 4.5 per cent. On the other hand, the bye-product of maize

manufacture contains 25 to 33 per cent. of dry substance, which almost entirely consists of digestible nitrogenous matters, whilst the residue of husks and shoots contains the greater quantity of the fat of the maize. The author goes on to consider the yield of starch, the exhaustion of the land, and the use of the nitrogenous matters as food bye-products. He compares the results with the cultivation of potatoes, and shews that the cultivation of maize in conjunction with the manufacture of maize-starch is the more remunerative. The bye-products repay the cost of working, so that the difference between the price of the maize and that at which the starch is sold is clear profit.—A. H. A.

*Action of Dilute Hydrochloric Acid upon Starch.* By F. Allihu. Dingl. Polyt. Journ. 250, 554.

THE object of the experiments detailed in this paper was to determine the conditions under which the maximum conversion of starch into sugar, is attained. The authors' results are given in the subjoined table. 12grms. of starch—containing 0.9 per cent. ash and 0.3 of insoluble residue—were treated in each experiment with 100c.c. of the boiling dilute acid.

Duration of Boiling Minutes	Percentage of Starch converted into Sugar by boiling with the Dilute Acid containing Per-cent. HCl.			
	10	5	3.33	2
2	92.6	—	—	—
5	92.4	—	—	—
10	—	90.6	—	—
15	91.7	—	—	—
30	89.6	94.3	93.27	84.91
50	87.1	93.3	—	—
60	—	—	91.65	93.68
90	—	—	94.19	95.05
105	—	—	—	94.89
120	—	—	—	93.81
150	—	—	—	94.65

The 2 per cent. acid is therefore to be preferred.—If it is desired to isolate the sugar the author recommends to recrystallise from methyl-alcohol, 0.816 specific gravity.

C. F. C.

*The Formation of Sugar in the Sugar Cane.* Aimé Girard, Compt. Rend., 97, 1305.

BY comparative investigations of the amount of cane sugar and grape sugar in different parts of the sugar-cane in the afternoon and before sunrise, the author has found that only in the substance of the leaves does this quantity vary, and that the quantity of cane sugar sinks during the night to one half, whilst the quantity of reducing sugar remains almost unaltered. He finds further that the quantity of cane sugar in the leaves increases with the illumination, on very bright days reaching nearly one per cent., considerably less on dull ones, and in either case diminishing during the night by one half. From this the author concludes that the formation of saccharose from glucose takes place entirely in the leaves under the influence of sun-light, and that the saccharose thereupon ascends the cane through the petioles, etc., and collects there.—A. S.

*The Extraction of Sugar from Molasses by Means of Lime.* Dingl. Polyt. Journ. 251, 313, 373, 415.

P. DEGNER has made exhaustive experiments on the recovery of sugar from molasses by lime and alcohol, and the results are published in the *Zeitsch. des deutschen Vereins für Rübenzuckerindustrie* 1883, p. 351 and 649. Details are given as to the preparation of the melassate of lime according to Scheibler-Seyferth's, Eissfeldt's, Weinrich's, Manoury's, and Drevermann-Sostmann's elution processes, the main features of which are that in Scheibler-Seyferth process finely-ground quicklime is added, in rather more than the theoretical quantity, to the molasses, with the result that the lime slakes at the expense of the water in the molasses, and a dry, porous



melassate of lime is obtained. The temperature during the reaction ranges from 115° to 129°. In the Eissfeldt process the same quantity of lime is used as in the previous case, but it is first mixed with a specified quantity of water, and is added to the molasses whenever the lime begins to slake. After standing some hours the mass has a plastic consistency, but becomes brittle after treatment with alcohol. In this reaction the temperature varies from 70° to 75°. The lime is completely slaked in the Weinrich process before being added to the molasses, and the mixture is kept warm for some hours. When cold the melassate is brittle, and by elution assumes a sandy form; temperature about 100°. In Manoury's process the molasses is first prepared by boiling in vacuo with a small quantity of lime; then 1.5 per cent. of soda is added, and the boiling continued, and the lime, after being slaked and sifted, is added, and the melassate formed in the usual way; temperature, 20° to 30°. In Drevermann-Sostmann's "precipitation" process, lime and 35 per cent. alcohol are added simultaneously to the molasses, the mixture is frequently stirred, and after 24 hours a fresh quantity of lime and alcohol is added, and after another 24 hours the elution takes place; temperature, 15° to 20°. The results of the experiments are given in 24 tables, from which it is seen that the mode of preparation of the melassate of lime does not materially affect the degree of purity of the saccharate. As the molasses takes up varying quantities of lime, there is no doubt but that this is accompanied by the separation of non-sugary matter, in the form of difficultly soluble lime salts. It is also very certain that an elevated temperature during the reaction favours the formation of organic lime salts. This formation in the Scheibler-Seyferth process is also aided by the lime, as it slakes in the molasses, being in what may be regarded as the nascent state. This condition also exists in the Eissfeldt process, but the quantity of water used prevents the temperature from rising. In the Weinrich process there is no action of nascent lime on the molasses, but this is made up for by the higher temperature to which the mixture is exposed. The temperature of melassates prepared according to Manoury and Drevermann is low, but this is partially compensated for by the larger number of points of attack for the lime in the former case, and the lime being distributed in the liquid in the latter. The following figures bear out the foregoing remarks. By the action of lime on molasses, on 100 parts sugar, were formed:—

	Parts organic lime salts.
Manoury .....	0.435
Eissfeldt .....	0.553
Weinrich .....	0.613
Scheibler-Seyferth .....	1.259

These results show the injurious action of temperatures over 100°. The next remarkable phenomenon in the preparation of melassates of lime is, that the evolution of ammonia is much smaller, even in the Weinrich and other processes, than in the Scheibler-Seyferth; perhaps, less on account of the production of smaller quantities than owing to the more perfect dessication by the superheated steam. Taking all things into consideration, it appears that the action of the lime in the molasses is not such a fruitful source of organic lime salts as was formerly supposed, the main cause of their formation being in the decompositions which take place during elution. By the action of lime on molasses, in all the processes, a less basic saccharate is always produced along with the tri-basic saccharate. This reaction depends on the quantity of water present, and the experiments show that the loss of sugar, owing to imperfect combination, is greatest in those processes which produce a melassate containing least water. Scheibler has shown that tri-basic saccharate of lime is practically insoluble in, and is not decomposed by, 35 per cent. alcohol, on the other hand the less basic saccharates are more easily dissolved. Consequently, when less basic saccharates are present, they are partially dissolved in the alcoholic solution, and, coming in contact with the soluble organic alkali salts, undergo double decomposition, producing insoluble organic lime salts, and saccharate of an alkali. This is also one of the causes of loss of sugar. There are thus

two undisputed causes of the formation of lime salts; the decomposition of organic alkali salts by saccharates, and by caustic lime. The mechanical and chemical structure of the melassate of lime has a material influence on the loss of sugar and production of lime salts. The quicker the alkali salts are removed during elution, the less chance will there be of forming insoluble lime salts, consequently the melassate which offers the greatest surface to the action of the alcohol will be best and quickest washed. Still it is noteworthy that a spongy porous melassate is not the best, because the pores serve as centres of chemical action, and soon become choked with lime salts. The fact that in all the processes the alkali salts can be nearly all removed, and still differences in the quotients of purity arise, is explained by the greater part of the non-sugary matter in the melassate being combined with lime during the elution, such non-sugary matter as it existed in the molasses being soluble in dilute alcohol, because it was combined with potash. That process is the best which most completely avoids the production of insoluble lime salts, both in the preparation of the melassate, and also during elution. Manoury tried to avoid, or at least diminish, the quantity of insoluble lime salts, by boiling the molasses with lime first, then decomposing the lime salts which have been formed, by carbonate of soda, thus producing carbonate of lime and organic alkali salts. These organic alkali salts, however, are soluble, and consequently can be washed out of the melassate, whereas had the non-sugary matter been in combination with lime, it would have been insoluble in the dilute alcohol, and have contaminated the sugar after saturation. This method does not entirely prevent the action of the lime on the non-sugary matter, but considerably restricts it. The loss of sugar is mainly caused by imperfect combination of the sugar with the lime in the melassate. Experiments prove that the smallest loss of sugar occurs in the melassate made by Eissfeldt's process. By too much washing the quotient may be improved, but the quantity of sugar diminished. The impurity of the saccharate caused by the decomposition of the saccharate of lime by organic potash salts is unavoidable in all the processes, but the further production of lime salts by the action of the excess of caustic lime in the melassate, on the potash salts, depends on the rapidity of the elution. The potash salts being much more soluble in dilute alcohol than caustic lime, it is possible to remove them before an appreciable quantity of lime is dissolved. In the first portions of the leys the largest quantities of sugar and non-sugary matter are found in the following order:

Non-sugary matter	Sugar
Eissfeldt	Manoury
Manoury	Weinrich
Weinrich	Scheibler-Seyferth
Scheibler-Seyferth	Eissfeldt
Drevermann	Drevermann

For dissolving approximately equal quantities of non-sugary matter, at the beginning of the elution, were necessary:

	Parts Ley
Manoury .....	1
Eissfeldt .....	1.18
Weinrich .....	1.81
Scheibler-Seyferth .....	4.01
Drevermann .....	8.18

The quantity of sugar dissolved therewith amounted to:

Eissfeldt .....	1.0
Weinrich .....	2.79
Manoury .....	2.89
Drevermann .....	3.20
Scheibler-Seyferth .....	3.96

The first leys also contained the largest quantities of free potash and here the largest quantities of lime salts are produced. But owing to difference in solubility between the potash salts and saccharate of lime, the first leys also contain the greatest amount of undecomposed alkali salts; the latter gradually disappear in the later leys as they contain enough lime and saccharate of lime to decompose the alkali salts still in solution. Finally there are no

more potash salts present in the leys, all the potash existing in the caustic state, and the organic alkali salts disappear from the leys in the following order:

Manoury  
Eissfeldt  
Weinrich  
Drevermann  
Scheibler-Seyferth

The employment of leys, instead of fresh alcohol, for elution, more completely utilizes the alcohol, but does not effect any saving in time, but the loss of sugar is diminished, and the leys, having a less solvent action on lime, the formation of lime salts is thereby hindered. The relation of the lime to the sugar in the different saccharates was for 1 molecule sugar, in the

Scheibler-Seyferth process .....	3.63 to 3.75
Eissfeldt process .....	2.70 to 3.04
Weinrich process .....	3.06 to 3.89
Manoury process .....	15.50 to 18.85
Drevermann-Sostmann .....	2.87 to 2.91

These figures show that the theoretical quantity were present in the Eissfeldt and Drevermann-Sostmann saccharates. The Scheibler-Seyferth saccharate was richer in lime, owing to the greater loss of sugar it had sustained. In Drevermann-Sostmann's process the small quantity of lime salts in the saccharate of lime is very noticeable. The agitation of the mixture of lime, molasses, and alcohol favours the rapid formation of a trisaccharate, and avoids the production of less basic saccharates. The combination of sugar with lime to an insoluble saccharate is more complete in this process than in any of the others. Gundermann proposed to partially purify the molasses, before the Drevermann process, by the addition of chloride of calcium and caustic lime, assuming rightly that by the conversion of a portion of the alkali salts into lime salts and chlorides of the alkalis, part of the non-sugary matter would be rendered insoluble in dilute alcohol, but this process is not a practical success owing to the difficulty in regulating the quantity of chloride of calcium for each case. The saccharate of lime obtained, after the molasses had been preliminarily treated by this process, contained more lime salts than that prepared in the ordinary way. The simplicity, rapidity, and completeness of the separation of the sugar in the manner first recommended by Drevermann must always be acknowledged.

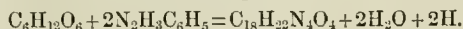
In the substitution process for the production of a tribasic saccharate, by lengthened stirring of a solution of molasses and milk of lime, a soluble monobasic saccharate is formed, which, on being boiled, deposits part of the sugar as tribasic saccharate. According to E. O. v. Lipmann (*Zeitsch. d. Vereins für Rübenzuckerindustrie*, 1883, p. 651, 880), it was found that by introducing finely-divided lime into a sugar solution, an immediate combination of sugar and lime took place, the same saccharate being found as that obtained in the substitution process. From this basis Steffen has discovered a new reaction and developed a new process, to which he has given the name "Ausscheidung." In order to completely precipitate the sugar from a solution, it is necessary to employ a solution of sugar saturated with lime, containing at the least 33 parts dissolved lime to 100 parts sugar. On adding a fresh quantity of lime to this solution, the sugar is immediately precipitated as granular tribasic saccharate of lime. The saccharate can be easily separated from the mother liquor by filtration, and by washing can be obtained almost pure. Tribasic saccharate of lime obtained in this way has the property of dissolving easily in sugar solutions not saturated with lime. At the same time, when proper proportions are used, the portion of lime not essential to the structure of the saccharate remains undissolved, and can be separated by filtration. W. Wolters (*Ger. Pat.* 2546, June, 1883) describes a "process for the recovery of sugar from aqueous solutions as tetra-basic saccharate of lime." Lime free from hydrate is employed, and preferably when it is over burnt. It must be in the form of a powder, but as moisture is always absorbed during the grinding, it is better only to break the lime coarsely at first, and finish the grinding in the sugar solution. A soluble tri-saccharate is first formed which, after standing

some time, combines with another molecule of lime and precipitates as tetra-basic saccharate, which can be separated from the liquor by filter process. According to instructions given by Wolters (*Deutsche Zuckerindustrie*, 1883 pp. 695) for the production of tetra-basic saccharate of lime, it is necessary to keep the temperature from rising, when mixing the lime and molasses. Over-burnt lime is coarsely ground in a mortar, and a quantity of an 8 or 10 per cent. solution of sugar, corresponding to one molecule of sugar to 3 molecules of lime, is poured on the lime, and then the grinding is continued; and, after filtration, the solution contains 1 molecule of sugar to 3 of lime. In the same way, 0.5 molecule of lime is again added, and, after a second filtration, a precipitate comes down consisting of 4 molecules of lime and 1 molecule of sugar.

W. M.

#### Compounds of Phenylhydrazine with Sugars. Emil Fischer. *Berl. Ber.* 17, 579.

PHENYLHYDRAZINE, according to the author's experiments, combines with all the sugars which reduce Fehling's solution, and the compounds formed being sparingly soluble in water, and therefore easily isolated, may be used for the detection and identification of sugars. The products are obtained by adding phenylhydrazine hydrochloride with excess of sodium acetate to an aqueous solution of the sugar, and heating the mixture on a water bath. In the case of dextrose, for example, if 1 part of the sugar be heated on the water bath with 2 parts of phenylhydrazine hydrochloride, 3 parts of sodium acetate and 20 parts of water, the separation of a precipitate, consisting of fine yellow needles, begins in ten or fifteen minutes. The precipitation continues until in an hour and a half the quantity of precipitate is equal to 85 or 90 per cent. of the dextrose used. The compound is almost insoluble in water, but is somewhat easily dissolved by boiling alcohol, from which it crystallises well on addition of water. The crystals melt at 204° to 205°, and on further heating decompose with carbonisation. Analysis leads to the formula  $C_{18}H_{22}N_4O_4$ , and the following equation may represent the chief reaction that takes place:—



The constitution of this substance, to which the name "phenylglucosazone" is assigned, has not yet been ascertained. By means of the above reaction one part of dextrose in 500 parts of water may be readily detected. It is proposed to utilise the test for the detection of sugar in urine. Laevulose, galactose, and sorbin behave in a similar manner to dextrose. Cane sugar is partially inverted, and then gives phenylglucosazone, whilst milk sugar combines directly with the reagent giving phenyl-lactosazone. Inosite and trehalose give no reaction.—A. S.

#### XVI.—BREWING, WINES, SPIRITS, Etc.

*Process of and Apparatus for Obtaining Extract from Hops to be used for Brewing.* E. Edwards, London. Communicated by G. Heller, Prague. *Eng. Pat.* 3954, Aug. 15, 1883. Provisional protection granted only.

By this invention hops are treated by steam at a temperature of 248° F. in order to obtain an extract from them, for the purpose of brewing beer. The apparatus consists of a cylindrical boiler, provided with a perforated bottom and an upper chamber communicating with the boiler by means of a valve suitably loaded and proportioned to blow off when steam of a temperature of 248° F. is introduced into the boiler. A pipe from this chamber communicates with a refrigerator or condensing vessel. A third vessel is also provided in which a portion of the hops is separately treated, this vessel consisting of an inclined perforated cylinder communicating by means of pipes with the steam space of the boiler on the one hand and the condensing apparatus on the other. The process described is as follows: Four-fifths of the hops are treated



in the boiler by steam which as described blows off when at a temperature of 248° F. and is condensed, together with the volatile constituents of the hops carried over, in the refrigerator. After about one hour and three quarters all the extractive matter will have been carried over; steam is then suddenly blown through the remainder of the hops placed in the before-mentioned perforated cylinder, only the finest flower perfume being thus carried over into the condenser. The contents of the condenser are then ready for use with the worts, into which they are discharged five minutes before the conclusion of boiling. The partially exhausted hops in the perforated cylinder are used in a succeeding operation in the boiler.—C. C. H.

*Treatment of Hops for the Obtainment of certain Useful Products.* William Linden, Streatham, Surrey. Eng. Pat. 3928, Aug. 13, 1883.

FRESH green hops are first submitted to a process of distillation in a cylindrical steam-jacketed vessel at a temperature of 125° C. to 175° C., steam also being admitted into the interior of the vessel; the essential oil passes over and may be collected from the distillate. After drying the hops are treated in the same apparatus with bisulphide of carbon at a temperature of 40° C.; the whole of the substances soluble in this menstrum are removed by successive operations, and the solution subsequently distilled to expel the carbon bisulphide, the residue consisting of the lupuline, myricine, and resin present in the hops. Treatment with alcohol effects the solution of the lupuline and the resin; their solution is filtered off from the myricine, and the alcohol distilled off. The residue is heated with a solution of potash or soda sufficient in quantity to convert the whole of the lupuline into lupulate of potash or soda, but a portion only of the resin into resinate. The residual hops are boiled with distilled water to dissolve out the tannin, the solution of which is evaporated to an extract. The essential oil, the alkaline lupulate and resinate, and the tannin can then be used for the preservation and flavouring of beer in a more economical manner than the usual method of using the hops in brewing. A further part of the invention provides for the production of pure lupulate of potash and soda for medicinal uses. The mixed residue of lupuline and resin, from the complete distillation of the bisulphide of carbon, is dissolved in ether; this is shaken with a solution of either potash and soda, the aqueous solution containing the resin being removed. The ethereal solution is then treated with distilled water, which dissolves out the lupulate of potash or soda. C. C. H.

*Treatment of Hops for the Purpose of Obtaining an Extract therefrom.* J. H. Johnson, London. Communicated by L. Bonté, Paris. Eng. Pat. 4151, Aug. 6, 1883.

IN order to obtain an extract of hops which meets the requirements of the brewer, and at the same time avoids the deterioration and disadvantages which result from the methods for this purpose hitherto employed, this invention proposes to effect the desired object: (1) by the mechanical separation of the lupuline from the hops; (2) the extraction of the fixed matters contained in the bracts or leaflets thereof. In order to effect this the hops are dried, so as to render them brittle, at a temperature of about 20° C., after which they are sifted on suitable sieves, the lupuline, together with broken portions of the bracts, passing through the sieves. This is set on one side, the fragments of the leaflets serving to prevent the volatilisation of the aromatic constituents. The sifted hops are then treated by lixiviation until their extractive matter is exhausted, the solution being rapidly evaporated *in vacuo* to a dry extract. This extract is then mixed with the lupuline previously obtained, and the whole stored in air-tight cases. The compound extract so obtained is preferably mixed in the proportion

desirable with the boiling worts about five minutes before running off the latter. The advantages claimed are facility of storage and transport, the better preservation of the aromatic properties, and the production of a well-flavoured beverage with more certainty and facility than heretofore.—C. C. H.

*The Determination of Tartaric Acid in Wine.* R. Kayser. Zeit. Anal. Chem. 23, 28—29.

ALL methods at present in use for determining tartaric acid in wine depend on the precipitation of the acid as the acid potassium salt in presence of alcohol, or ether and alcohol, and titration of the precipitate with decinormal soda solution. In addition to the known errors of this method, the author points out another, due to the precipitation of a portion of the acid as the neutral calcium salt, which is not affected by the standard soda solution. The error produced is considerable, since one part of lime combines with 2.68 parts of tartaric acid; it can, however, be calculated and allowed for by estimating the lime in the precipitated tartarates. In fourteen determinations on various wines, the average percentage of tartaric acid was 0.077, and the average percentage of acid thrown down as calcium tartrate was 0.031.—H. B.

## XVII.—FOOD PRESERVING, MEATS, Etc.

*The Employment of Boracic Acid in the Preservation of Foods.* J. Forster. Dingl. Polyt. Journ. 251, 170, 1884.

IN attempting to form an opinion as to the value of certain antiseptic substances for the preservation of foods two important questions present themselves. First, to what extent can such substances be employed without producing the effects of medicinal doses; secondly, supposing the diet to be composed entirely of such preserved food, is there any limit to the percentage amount of added antiseptics which may not be passed if a normal action of the alimentary organs is to be insured. The author has made the two following series of experiments with boracic acid. In the first series, which extended over a space of three days, he observed the influence of small doses of boracic acid taken with a mixed diet which daily consisted of 475.7 grms. of total solid matters, 17.33 grms. of nitrogen, 140.4 grms. of fat. In the second series the diet was entirely composed of milk and eggs. Of the solid matters and nitrogen which were passed into the alimentary canal the percentage amounts here given were not absorbed but were lost in the feces.

### FIRST SERIES.

	Dry Substances.		Nitrogen.	
	%		%	
Experiment 1 (without boracic acid)....	12.4	20.0	2.4	20.0
Experiment 2 (with 3 grms. boracic acid)....	14.8	23.9	2.8	23.9
Experiment 3 (without boracic acid)....	14.2	22.5	2.2	22.5

### SECOND SERIES.

Experiment 1 (with 1.5 grm. boracic acid)....	6.2	4.2
Experiment 2 (without boracic acid)....	5.2	3.1
Experiment 3 (with 0.5 grm. boracic acid)....	5.8	4.0
Experiment 4 (without boracic acid)....	4.6	3.0

From these two series of experiments it is apparent that boracic acid, in quantity too small to have any ordinary therapeutical action, has such an influence on the digestive organs as to cause an increase in the feces excreted, and to raise the proportion of solid matters and nitrogen contained therein. It is also noticeable that the extent of its action is proportional to the quantity received into the system, and that even so small an amount as 0.5 grm. produces undoubted effects. Not only do these results occur with a mixed diet, containing a certain proportion of indigestible constituents, but likewise with a diet of milk and eggs, which, according to many experiments, are almost entirely absorbed under ordinary circumstances. As to the reason for this increased excretion, two views may be taken. First, it is possible that under the influence of boracic acid there is an increased secretion of digestive liquids, the solid matter contained in which would be added

to the faeces. This view of the case is not unfavourable to the employment of boracic acid. According to the second view, this increased excretion is due either to a decreased absorption into the system of food substances or to an abnormal separation of epithelium and such materials of the mucous membrane. In support of the first view it may be adduced that faeces from milk preserved from boracic acid contain a larger amount of fatty acids and soluble nitrogenous matters resulting from the decomposition of bile than the faeces from normal milk; but, on the other hand, the second view is supported by the fact that faeces from milk preserved with boracic acid contain a larger proportion of insoluble nitrogenous matters than the normal. To whatsoever secondary cause this excretion of nitrogenous matters is to be ascribed, the primary influence of boracic acid is certainly of greater importance than has been hitherto suspected, and it will be desirable generally to observe caution in its use, and especially in dieting young children on milk preserved by its agency.—W. D. B.

*A New Process for the Elimination of Nitrogenous Matters from Fermentable or Fermented Substances.* E. R. Moritz and H. C. Lee, London. Eng. Pat. 3536. July 18, 1883.

HITHERTO the elimination of nitrogenous matters, which act as nutriment for the germs by which putrefactive or secondary fermentive changes are caused, has been attempted by addition to the fermented or fermentable substance of antiseptics in order to destroy these germs. The objections to this method are that the embryo germs cannot thus be destroyed, and the antiseptic imparts disagreeable properties to the substance treated. The new process consists in precipitating the nitrogenous matters which form the germ food by means of phosphotungstic acid and its salts, the precipitate being afterwards removed by mechanical or other known means.—A. W.

*Improvement in the Method of Preserving Fermented Liquors.* R. G. Bell, Oxford. Eng. Pat. 4473. March 6, 1884.

THE preservation of fermented liquors in good condition has hitherto been effected either by heating to a temperature of from 130° to 155° F. in the vessels in which they are contained, or by the injection of carbonic acid gas. The patentee in this case claims the combination of these two methods, the injection of the carbonic acid gas under pressure in conjunction with heating up to the temperature before stated. The ordinary apparatus now in use for each purpose may be employed, and either one or the other may be performed first, or both operations may be concurrent with each other.—C.C.H.

## XVIII.—SANITARY CHEMISTRY, DISINFECTANTS, Etc.

*Are Arsenic Compounds Poisonous for Vegetable Proto-plasm?* O. Loew. Central-blatt. f. Agrikulturrehmie. 13, 68.

THE acids of arsenic are poisonous towards algae only in the same degree and for the same reason as are acetic and citric acids. On the other hand, *Spirogyra* thrive in spring water containing one-thousandth of potassium arsenate. The larvae of insects and infusoria can also live for weeks in such a water, but water snails and beetles die in from one to two days.—H.B.

## XIX.—PAPER, PASTEBOARD, Etc.

*A. Mitscherlich's Improvements in the Manufacture of Wood Pulp.* Dingler 251. Heft. 6.

THE figure (Fig. 2) shews part of the boiler with the inner coating and manhole, the dimensions being 4m. in

diameter and 12m. in length, thus allowing considerable quantities of wood to be worked at a time. The inner coating of the boiler A is formed by a covering of lead  $a_1$ , which is fastened by means of a cement of tar and pitch. This is warmed, and the thin leaden sheets are pressed down on to the iron, thus forming an efficient protection against the action of acids. Should the lead give way at any point, all that requires to be done is to warm the damaged part and press a sheet of lead over the burst place. Even in a case where the connecting cement under the lead was ruptured, the action would only be local, and could not extend along the iron. The leaden coating is again protected by a coat of cemented tiles of a porcelain-like material. The lead used should be thin, so as to enter into all depressions and be the easier joined to the iron by the pitch cement. If the lead be applied as above,

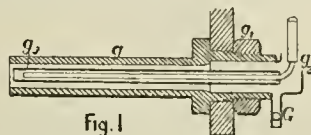


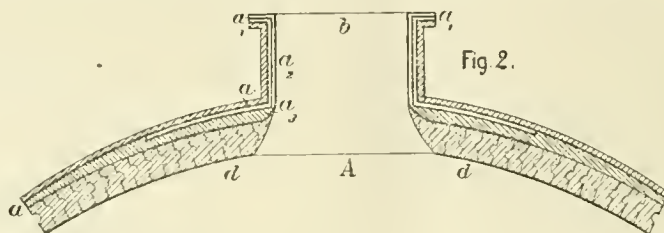
Fig. 1

the costly burning of each single plate is avoided. The manholes  $b$ , used for filling and emptying the boilers, have another lead covering  $a_1$  besides the first one  $a$ . This second lead is likewise fastened by the tar and pitch cement. Further, there is a third covering  $a_2$  also of lead, which extends into the boiler over a very thick layer of cement  $a_3$ . These various coatings of lead and cement are necessary, since the manholes, of course, cannot have the protection afforded by the glazed tiles  $d$ . When the leaden layer  $a_2$  is worn out it can be removed and replaced by a new one. A system of tubes heats the boiler, and these cover the lower half of the same from the bottom up to about the middle. A large number of tubes heated by steam are used, since in case one becomes defective the same can be shut off and the boiling process need not be interfered with. The tubes should be made of an alloy of Pb and Sb, so as to insure the maximum available resistance to chemical and mechanical action. Both ends of each tube are fastened to the wall of the boiler by an arrangement shown in Fig. 3. The short thick leaden pipe B is attached to the boiler plate A by means of the washer  $e$  and the nut  $e_1$ . The piece  $f$ , which has been slipped over the tube R, is screwed into B; an annular washer  $i$  of lead or asbestos is then passed over the tube and screwed tight by the nut  $e_1$ . In this manner a steam-tight and acid-proof connection of tube and boiler is attained. For testing the contents and the easy observation of temperature and pressure, the arrangement shown in Fig. 1 is employed, and this only requires a single opening in the boiler plate. The tube  $g$  is fastened by the nut  $g_1$  to the boiler plate, and carries the thermometer  $g_2$ , which is further protected by the tube  $g_3$ . Between  $g$  and  $g_3$  sufficient room is left to allow a small portion of the contents of the boiler to be withdrawn by the valve G. A manometer and also a gauge glass, to shew the height of fluid in the boiler, are attached. After a sufficient amount of boiling, the wood goes to the stamping mill, where the fibres of the pulp are separated and the incrusting substances washed out. In order to avoid the reduction of the knots, the hammers do not touch the bottom of the trough, and so they principally act by pressing, and the knots are not pulverised. In boiling the wood the following are the chief points. The bark is removed, the wood cut into pieces and then steamed in order to prepare for the subsequent action of the acid and to remove air as much as possible, which facilitates the entrance of acid, etc., into the pores. During this operation the temperature must not rise above 100° C., since otherwise the steaming process is not so advantageous. After steaming comes the boiling with sulphite of lime. The magnitude of the charge and the concentration of the liquor must stand in a certain definite relation, otherwise insoluble salts are formed on the wood fibre, and these can only be washed out with difficulty. Such stuff when worked into paper will invariably come out knotty. Besides this the cost of bleaching is then much increased, as more bleaching powder must be

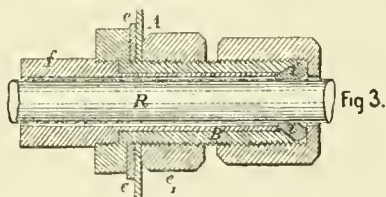


used. If the charge of wood be too heavy, then the process remains incomplete. Occasional tests are made with small quantities which are periodically withdrawn. During the boiling process, time and temperature must be well watched. The first stage consists in a slow action, and best takes place at a temperature of  $108^{\circ}\text{C}$ . The second state is more energetic, and requires a temperature

up to  $118^{\circ}\text{C}$ ., gradually attained. On boiling off the  $\text{SO}_2$  the same effect is attained as by reducing the temperature. The testing as to the completion of the process is carried on as follows: Addition of ammonia in excess will throw down a precipitate, the smaller in volume as the boiling approaches completion. If the precipitate be about  $\frac{1}{10}$ th of the volume of the fluid taken, then the time has arrived



for boiling off the  $\text{SO}_2$ ;  $\frac{1}{2}$ nd volume shews that the boiling is finished, and the solution requires to be run off rapidly. A smaller precipitate shews that the boiling has been carried on too far, and that no active agent is any longer present. In this case free  $\text{H}_2\text{SO}_4$  would impart a detrimental brown colour. For a safe boiling process to be carried on smoothly through the various stages, it is advantageous to have a large boiler fitted as above described. A high temperature would indeed quicken the process, but would also require higher temperatures, and the product would be found of lower quality and less in toughness and quantity. As to chemical reactions, we



have the following: On boiling the  $\text{SO}_2 \cdot \text{H}_2\text{O}$ , a portion of the oxygen of the organic matters causes the formation of  $\text{H}_2\text{SO}_4$ , which under normal conditions combines with the bases which were in combination with the sulphurous acid. When the process is badly conducted free  $\text{H}_2\text{SO}_4$  is formed, and this more or less carbonises and discolours the fibres. Also the incrusting salts form compounds with tannic acid. An essential is the freedom of the sulphurous solution from polythionic salts, as these are found very injurious. These polythionates usually form through the presence of free sulphur vapour during the burning process.—H. A. R.

*Improvements in the Treatment of Straw, Straw Refuse, or other Fibrous Materials for the Manufacture of Pulp and Manure.* T. H. Cobley, Dunstable, Bedford. Eng. Pat. 3599, July 23, 1883.

To make pulp for the production of white or brown paper or board from straw, stable litter, or straw paper waste, etc., the material is first cut into inch lengths, and then macerated in a solution of calcium, magnesium, or other chlorides. It is next boiled with a liquor composed of lime and calcium chloride (for brown pulp) or caustic or carbonated soda or potash (for white pulp). The brown pulp is next washed, and acidulated with sulphuric or hydrochloric acid, and may be afterwards partially bleached by carbonate of soda. The white pulp is washed and treated with an acid solution of calcium or magnesium chloride, then bleached with chloride of lime or the mixed hypochlorites of potash and magnesia obtained from kainite by double decomposition with bleaching powder. The pulp is finally washed, squeezed through india-rubber rolls, and dried by hot air. When straw-paper waste is used maceration is not required,

When stable litter is used the macerating liquor must be stronger, and should contain chloride of iron. The liquor resulting from the maceration is precipitated by a salt of alumina, and the precipitated sludge pressed in a filter press and sold as manure with or without the addition of other fertilising matter. To size the paper or board leather waste or glue waste is boiled with an alkali, and a mixture of lime and alum used to precipitate the size on to the fibre. For dark or black board a salt of iron is added. The claims are—(1) for producing producing white pulp, (2) brown pulp, and (3) manure from the materials and by the means described.—J. M. H. M.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

*Jackson and Mencke's Method of Preparing Borneol from Camphor.* J. Kachler and F. V. Spitzer. Ber. 17, 141, and Monatsh. für Chem. 5, 50.

THE authors have attempted the preparation of borneol by reducing an alcoholic solution of camphor with sodium as recommended by Jackson and Mencke (Ber. 16, 2930), but have not succeeded. The product is a mixture of about 80 parts of camphor and 20 parts of borneol.—D. B.

*Synthesis of Piperidine.* Ber. Berichte 17 [2], 156.

THE author was unable to obtain piperidine from pyridine by the action of tin and hydrochloric acid (Königs, *ibid.* 14, 1856), but succeeded in effecting a partial conversion by the action of sodium upon an alcoholic solution of the base. The bases were separated by conversion into the nitroso derivatives, as suggested by Königs. The author is extending his investigations to the homologues of this series.—C. F. C.

*Hyoscine.* A. Ladenburg and C. F. Roth. Berl. Ber. 17 [2], 151.

THIS body was isolated by the authors from the residues from the preparation of hyoseyamine. It is resolved by the action of alkalis into tropaeic acid and pseudotropine, a base isomeric with tropine. Pseudotropine melts at  $106^{\circ}$ , tropine at  $62^{\circ}$ . It boils at  $241^{\circ}$  to  $243^{\circ}$ . It crystallises in rhombohedra, which are soluble in water and in chloroform. By the action of methyl-iodide upon an aqueous solution of the base at  $100^{\circ}$ , it is converted into the methyl-iodide,  $\text{C}_8\text{H}_{15}\text{N} \cdot \text{CH}_3\text{I}$ . On bringing this compound into contact with recently-precipitated silver chloride, the iodine is replaced by chlorine, and the chloride thus formed gives with  $\text{PtCl}_4$  the platino chloride ( $\text{C}_8\text{H}_{15}\text{N} \cdot \text{HCl}$ ) $_2\text{PtCl}_4$ , which crystallises in orange-coloured shining plates.—C. F. C.

*Adulteration of Tartar Emetic.* Dingler 251. Heft 6. H. SCHMID has shewn in the *Chemiker Zeitung*, 1882, p. 949, that in many cases zinc acetate can replace tartar emetic with advantage. This fact has since been made use of to adulterate the latter drug with the lower-priced zinc sulphate, and such mixtures have been introduced into commerce as "tartar emetic powder," "tartar emetic substitute," "antimony mordant," etc., etc. According to *Chemiker Zeitung*, 1884, p. 23, from 33 to 59 per cent. of  $\text{ZnSO}_4$  has been found in similar preparations. On making a proportional calculation based on the price of the mixture, and the analytical result, the price of each per cent. of antimony oxide, which alone is the valuable ingredient in tartar emetic, came out at 12s. 3d. for the sample, containing 59 per cent. of  $\text{ZnSO}_4$ , whilst in a 99 to 100 per cent. sample, viz., a commercially pure preparation, the price stood at 7s.—H. A. R.

*Belladonnine.* By A. Ladenburg and C. F. Roth. Berl. Ber. 17 [2], 152.

THE authors have investigated the action of alkalis upon this base. Amongst the products of resolution they identified tropine, an oxytropine, and tropaic acid. Further researches upon this base are in progress.—C. F. C.

*Derivatives of Pyridine.* W. Königs and R. Geigy. Ber. 17, 589.

PREPARATION of hydroxypyridinecarboxylic acid from hydroxyquinolinic acid. By fusing quinolinic acid with potash, Königs and Koerner obtained a hydroxyquinolinic acid, which they did not consider to be related to Lieben and Haitinger's ammonchelidonic acid, although the latter appears to be a hydroxypyridine dicarboxylic acid. Whilst ammonchelidonic acid when heated with water at  $195^\circ$  is resolved into carbonic anhydride and hydroxypyridine, hydroxyquinolinic acid when treated in an analogous way liberates only one molecule of carbonic anhydride, and is converted into hydroxypyridine carboxylic acid,  $\text{C}_6\text{H}_5\text{NO}_3$ . The latter separates from hot water on cooling in colourless crystals, melting at  $301^\circ$ – $302^\circ$ . It does not give a red colouration with ferric chloride. Its aqueous solution is not precipitated by calcium, lead and copper acetate and silver nitrate. A solution of its sodium salt gives precipitates with copper, lead, and silver salts. The acid obtained by heating hydroxyquinolinic acid with water differs from the three hydroxypyridine carboxylic acids hitherto obtained, which Ost and Bellman have prepared from comenamic acid, and regard as derivatives of picolinic acid. Hydroxypyridine,  $\text{C}_6\text{H}_5\text{NO}$ , is obtained by distilling the hydrogen silver salt of hydroxyquinolinic acid in a current of carbonic anhydride. It crystallises from benzene in white needles, melting at  $106^\circ$  to  $107^\circ$ . Its aqueous solution gives a red colouration with ferric chloride. On treating a concentrated aqueous solution of hydroxypyridine with bromine dibromhydroxypyridine,  $\text{C}_6\text{H}_2\text{Br}_2\text{NO}$ , is obtained which crystallises from hot water in long white needles, melting at  $206^\circ$  to  $207^\circ$ . It is readily soluble in alkalis, from which solutions it is precipitated unaltered on addition of acids. It is not identical with the dibromhydroxypyridine which Hoffmann obtained by the action of bromine on piperidine. The hydroxypyridine obtained from hydroxyquinolinic acid differs from the hydroxypyridine which Ost has recently prepared from comenamic acid, and which he considers to be identical with the product obtained by Lieben and Haitinger from ammonchelidonic acid. Pyridinedisulphonic acid. Hoffmann and Koenigs have recently referred to the formation of a disulphonic acid of pyridine by heating piperidine with concentrated sulphuric acid. They have isolated the barium salt which crystallises in white needles readily soluble in water. The free acid is insoluble in alcohol and ether, sparingly soluble in glacial acetic acid, and readily soluble in water. It crystallises from concentrated aqueous solutions, and is obtained in white needles by dissolving it in boiling acetic acid (50 per cent.) and adding glacial acetic acid. The authors have prepared the potassium, sodium, and lead salt, all of

which give well-defined crystals. The di-acid is distinguished from the mono-acid by its behaviour with bromine. By adding bromine to an aqueous solution of the monosulphonic acid it is resolved into dibromopyridine melting at  $164^\circ$ , the sulpho group being split up into sulphuric acid; whilst, by treating the disulphonic acid under similar conditions, sulphuric acid is formed, but only small quantities of a bromine compound. Phosphorus pentachloride does not act on the barium salt of pyridinedisulphonic acid until a temperature of  $200^\circ$  has been attained, when the formation of chlorinated pyridines takes place. The authors have prepared trichloropyridine  $\text{C}_5\text{H}_2\text{NCl}_3$  melting at  $48^\circ$  and crystallising in silky laminae. It is intended to study the products obtained by the action of phosphorus pentachloride, potassium cyanide and alkalis on pyridinedisulphonic acid at an elevated temperature.—D. B.

*Thebaine.* W. C. Howard. Berl. Ber. 17, 527.

By the action of fuming hydrochloric or hydrobromic acid on thebaine the author has obtained salts from which, by the action of alkalis, a new base is obtained. The formula of this substance is  $\text{C}_{17}\text{H}_{17}\text{NO}_3$ , and the name morphothebaine is given to it to express its probable close relation to morphine. The author has prepared salts, the acetyl derivative, and some addition compounds with the iodides and chlorides of the alcohol radicals.—A. S.

## XXI.—EXPLOSIVES, MATCHES, Etc.

*Experiments with compressed Gun Cotton.* By Max v. Förster, Berlin, 1883. Critically discussed by O. Guttman. Dingl. Polyt. Journ., 250, 466.

THE gun cotton used in these experiments contained 12.3 per cent. nitrogen. Air dry it contained 1 per cent. hygroscopic moisture. The experiments led to the following conclusions: (1) Cartridges of equal diameter but of varying heights, therefore of varying weights, exert approximately equal effects; (2) Cartridges with a hollow centre are more effective than solid ones of the same dimensions; (3) The effect is considerably increased by adding barium nitrate to the charge, and moist gun cotton is more effective than dry; (4) The larger the sectional area of the cartridge, the more widely spread its effect, the direction of propagation of which is moreover determined by the area of contact of the charge, i.e., the base of the cartridge; (5) In overcoming an external resistance the greatest effect is produced by starting the explosion on the side farthest removed from the object, or by hollowing out the cartridge on the side nearest to the object. In criticising these results, Guttman points out in the first place, that the author's experiments, in which the explosion took place in an open space, are useless as a basis of inferring what takes place in a confined space; and that they are fully explained or even anticipated by principles already established, notably Abel's theory of explosions.—Thus the first condition of the effect is the duration of propagation of the explosion; with the gun cotton under pressure, or in the moist state this is minimised, and the explosion being concentrated produces the effect proportionate to the weight of substance exploded. Förster's processes of rendering gun cotton and gun cotton cartridges waterproof, the first by immersion in ether, the solvent action of which upon the lower nitro celluloses causes the formation of a collodion skin, and the second by immersion in melted paraffin, are regarded by the author as ineffective, as points of entrance, however small, are left open to the water. Förster's inference from the observed degradation of gun cotton into acid gummy (pectic) bodies, that the spontaneous decomposition of the nitrocelluloses is never attended with explosion, the author regards as unwarranted.—C. F. C.

*Improvements in the Manufacture of Explosive Compounds for Blasting Purposes.* Silas R. Divine. Eng. Pat. 5625. Dec. 4, 1883.

THIS invention relates to a plan for increasing the susceptibility to explosion under various conditions of



confinement of an explosive substance consisting of a mixture of potassium chlorate with coal tar, "dead oil," oil of turpentine, nitro-benzene or non-volatile hydrocarbons as described in Eng. Pat. 5624, Dec. 4, 1884. This object is to be attained by the addition of nitro-glycerin or sulphur to the above described mixtures. In the case of nitro-glycerin being employed, this is mixed with such proportion of the liquid ingredient that the mixture contains not more than 60 per cent. and not less than 20 per cent. of nitro-glycerin, for when the latter limit is reached the sensitizing effect apparently disappears. Good results can be obtained by mixing 2 parts of "dead oil" with 1 part of nitro-glycerin and absorbing this mixture by 15 parts potassium chlorate; or 1 part of "dead oil," 1 part of nitro-benzene and 1 part of nitro-glycerin with  $13\frac{1}{2}$  parts of potassium chlorate. The increase or decrease of the nitro-glycerin in the mixture constituting the liquid ingredient respectively augments or diminishes the rapidity of combustion of the final compound hence the action of the explosive may be regulated at will. The liquid ingredient thus formed is comparatively insensible to friction or percussion and may be transported with safety. In the case of sulphur being employed this is dissolved in carbon bisulphide or gasoline in proportions such as will supply from 1 to 3 per cent. of sulphur on the total potassium chlorate present. To the quantity of the liquid ingredient ("dead oil," etc.), which is the proper proportion relatively to the solid ingredient (chlorate of potash), such a volume of the above sulphur solution is added as will make a total fluid volume equal to the full fluid-absorbing capacity of the solid ingredient. The union of the solid and liquid ingredients is effected as described in Eng. Pat. 5624, 1883, whilst the sulphur, on evaporation of the volatile solvent, becomes disseminated throughout the mass of the cartridge, which, being in a permanently moist condition from the presence of the "dead oil," is not so liable to accident as a dry mixture of sulphur and potassium chlorate would be. The explosive compound thus formed has an increased sensitiveness or susceptibility to explosion, but is comparatively insensible to percussion or shock. It requires to be fired with a detonator to obtain the best effects. By sprinkling sulphur over the envelope containing the chlorate of potash after immersing the former in the liquid ingredient, the same sensitising effect may be obtained as by the dissemination of particles of sulphur throughout the mass.—W. D. B.

*Experiments with Compressed Gun-cotton.* M. v. Förster, Dingl. Polyt. Jour. 251, 371.

The following is a reply to a criticism by Oscar Guttman, (Dingl. Polyt. Jour. 250, 456) of a pamphlet containing experiments made by the author on the best way of employing compressed gun cotton. The author can now better determine in how far similar conditions arise in the development of force when gun-cotton is detonated in closed and open spaces, and thinks that the hitherto existing theories and experiments do not fully explain this point, his experiments do not completely clear it up but throw a good deal of light on the subject. In reference to his process for coating pieces of compressed gun-cotton by dipping them in a solvent, he remarks that acetic ether, as well as nitrobenzol, and various other substances, dissolves gun-cotton in the same way as a mixture of alcohol and sulphuric ether dissolves collodion. His process differs from the numerous other patented methods for covering cartridges of gun cotton, dynamite, etc., with a waterproof coating of collodion, in that he dissolves the hitherto considered insoluble gun cotton, thus forming the coating of the substance itself. Gun-cotton which has been well nitrated contains 7 to 10 per cent. of collodion, and if a piece of such gun-cotton be dipped into ether alcohol, the solvent of collodion, the solution penetrates into the gun-cotton without forming a skin. On the other hand, gun-cotton, which has been freed from collodion, by extraction with ether alcohol, is completely soluble in acetic ether. The cause of the skin, produced by his process, not being quite water proof is that each piece of compressed gun-cotton has minute rents on the surface. These cracks, invisible to the naked

eye, in no way affect the stability of the pieces of gun cotton. To make them quite waterproof he now laquers the pieces which have already a firm skin. Concerning the spontaneous decomposition of gun cotton, he says that gun cotton even when badly made, under ordinary conditions will not decompose with production of fire, or even explosion. When good gun cotton is exposed for months to a high temperature ( $110^{\circ}$ ), it continues to loose nitrogen, until finally a brown powder is left. It never bursts into flame. Of course when the temperature is raised to the point of ignition (with good gun cotton and gradual heating,  $175^{\circ}$  to  $180^{\circ}$ ; sudden heating,  $240^{\circ}$ ; when the acid has been imperfectly removed it falls to  $140^{\circ}$ ), the gun cotton burns, but this is not spontaneous combustion. In the manufacture of gun cotton, after being washed until it does not redden litmus paper, it is still regarded as being imperfectly washed, and it is not until Professor Abel's process, of reducing the fibrous cotton to dust and then washing it again, has been employed, that a perfectly washed article is obtained. Formerly before this process was used, many samples of impure gun cotton may have got into use, but the author has never heard of an authenticated case in which even such gun cotton took fire or exploded by spontaneous decomposition, and he would be very glad to receive any information of such a case having occurred.—W. M.

*Improvements in the Manufacture of Explosives, chiefly for Blasting Purposes.* Silas R. Divine. Eng. Pat. 5624, Dec. 4, 1883.

THIS invention relates to the preparation of an explosive from two inexpensive ingredients—the one a solid, the other a liquid, which are to be mixed as required. The operation of mixing is without danger, and may be performed without skilled labour. The solid ingredient consists of chlorate of potash, for which, however, the permanganate or perchlorate may be substituted. The liquid ingredient may consist either of the heavy oil of coal-tar known as "dead oil," of a mixture of equal parts of nitro-benzene and dead oil, of oil of turpentine or of fluid hydrocarbons not volatile at  $300^{\circ}$  F., or of such hydrocarbons previously treated with nitric acid. The most advantageous proportions are  $7\frac{1}{2}$  parts of chlorate of potash to 1 part of dead oil; from 8 to 10 parts chlorate of potash to 1 part of dead oil, mixed with 1 part of nitro-benzene; and  $8\frac{1}{2}$  parts of chlorate of potash to 1 part of oil of turpentine—proportions which may be slightly modified according to the state of oxidation of the turpentine. To effect the mixture of the solid and liquid ingredients, cartridges of the required diameter, formed by inclosing the solid ingredient chlorate of potash in porous envelopes of cloth or paper, are immersed in a bath of the liquid ingredient for from three to six seconds, according to their diameter, removed, and a superficial excess of liquid allowed to drain off. After standing for a short space of time, the absorbed liquid becomes equally distributed through the entire mass of the cartridge, which is then ready for use, and may be fired by either detonator or fuse. The mixture of the solid and liquid ingredients may also be effected by dissolving the liquid constituent in an equal bulk of a volatile liquid, such as gasoline or carbon bisulphide, and immersing the cartridge of the solid ingredient in this solution until it has absorbed its maximum amount. On removing the cartridges from this solution, and permitting the volatile fluid to evaporate spontaneously the explosive is obtained ready for use. The cartridges may be at once seated in drill holes in rock, confined by tamping and fired by a detonator.—W. D. B.

*Improvements in the Manufacture of Gunpowder.* Owen Bowen. Eng. Pat. 3876. August 9th, 1883. Provisional Protection only.

POWDERED lignite which has been carbonised to remove the water which it contains is mixed with sulphur and nitre in the following proportions: viz., lignite 18 to 21 per cent. sulphur, 4 per cent., nitre 74 to 77 per cent., adding about 1 per cent. of water during the mixing to ensure the coherence of the particles. By thus reducing

the amount of sulphur as compared with that present in ordinary gunpowder, it is claimed that a reduction of the smoke, and erosive effect upon the bore of the gun, is obtained, whilst the powder being of slow combustion an excessive strain is not likely to be generated. The calcined lignite is less hygroscopic than charcoal and thus the excess of the lignite will not have the deleterious effect that an excess of charcoal usually has.—W. D. B.

## XII.—ANALYTICAL CHEMISTRY.

*Estimation of Potassium Iodide.* Dingl. Polyt. Journ. 250, 469.

KASPAR'S method of estimating potassium iodide by titration with a standard solution of mercuric chloride (ibid. 244, 255) has been shewn by Leuz (Zeitschr. F. Anal. Ch., 1883, 391) to be inaccurate by reason of the influence exerted upon the reaction, by the temperature of the solutions. P. Carles, who has also investigated the method, finds that further variations are caused by variations in the degree of dilution of the solution, and on the basis of a careful series of experiments, pronounces it untrustworthy.—C. F. C.

*New Method for the Qualitative Separation of Tin, Antimony and Arsenic.* E. Berglund. Berl. Ber. 17, 95.

THIS method depends upon the fact that when a solution of the three sulphides in sodium sulphide is boiled with cupric oxide, stannate, antimoniate, and arsenate of sodium are formed. Cupric oxide in a state suitable for the purpose is obtained by igniting the nitrate and reducing the resulting oxide to a fine powder. After use and partial conversion into sulphide, it can readily be reconverted into oxide by ignition. The process is as follows: The mixed sulphides obtained in the ordinary course of analysis dissolved in ammonium sulphide, are precipitated by hydrochloric acid, and after washing, dissolved in a boiling solution of sodium sulphide. Cupric oxide is then added and the boiling continued until (after the lapse of a few minutes) it rapidly subsides, leaving the supernatant liquid colourless. The liquid is at once filtered, and after cooling the filtrate, mixed with  $\frac{1}{4}$  its bulk of alcohol. The sodium antimoniate thus separates out as an extremely fine powder, which is very difficult to filter off, and to the filtrate after boiling to expel alcohol, is added an excess of ammonium chloride. This precipitates the tin, and the arsenic wholly or partially as  $2\text{SnO}_2 \cdot \text{As}_2\text{O}_5$ . A few drops of ammonia are now added, and sulphuretted hydrogen passed into the solution until it is clear. The arsenic is precipitated as magnesium ammonium arsenate, by the addition of  $\frac{1}{3}$  the volume of ammonia and magnesium mixture, and after standing an hour filtered off. On acidifying the filtrate with hydrochloric acid, tin is precipitated as  $\text{SnS}_2$ . The author proposes to ascertain the possibility of applying this method quantitatively.—A. S.

*Phenylhydrazine as a Reagent for Aldehydes and Ketones.* Emil Fischer. Berl. Ber. 17, 572.

FROM a series of experiments made on the subject the author recommends the use of phenylhydrazine  $\text{N}_2\text{H}_3 \cdot \text{C}_6\text{H}_5$  as in many cases preferable to hydroxylamine for detecting and distinguishing aldehydes and ketones on account of the easy manipulation. The base, moreover, is very readily prepared. The reagent is prepared as follows: Phenylhydrazine hydrochloride is obtained by neutralising distilled phenylhydrazine dissolved in 10 parts of alcohol with concentrated hydrochloric acid, the crystalline mass which separates out, being washed with alcohol and ether till colourless, and dried on a water bath. One part of this and one and a half parts of sodium acetate are then dissolved in 8 to 10 parts of water. In applying the reagent an aqueous solution of the aldehyde or ketone (even when these are only slightly soluble in water) may in most cases be used, otherwise alcohol is employed. The reagent is added in excess to a cold aqueous solution of the aldehyde or ketone when at once or after a short time (depending on the concentration of the solution) the

condensation product separates out as an oily or crystalline precipitate. Before applying the test free mineral acids must be neutralised by caustic soda, and if nitric acid is present it had better be eliminated by previous addition of urea. In the case of many complicated aldehydes and ketones, for example those of the sugar class, the reaction must be facilitated by heat. If the condensation product obtained as above is solid, a determination of its melting point is usually sufficient to identify it, and therefore also the aldehyde or ketone under examination. If the product is oily the test is less distinctive; it affords nevertheless a means of proving the presence or absence of a ketone or aldehyde. Acetic, propionic, butyric, valeric aldehydes and cinnamaldehyde give colourless non-crystallisable oils difficult of identification. Furfural on the other hand is very readily detected, one part dissolved in 10,000 of water giving a characteristic crystalline precipitate in fifteen minutes. Benzaldehyde, cinnamic aldehyde, salicylic aldehyde and glyoxal among the aldehydes are also readily detected. The ketones of the fatty series give oily condensation products not easily recognised, but affording a convenient means of separating the ketones from other substances. Acetophenone, benzylideneacetone, benzophenone and isatin on the other hand give characteristic products. The test is also applicable to ketonic and aldehyde acids, such as glyoxylic acid.

A. S.

*The use of ferric sulphate for the detection of Iodides in presence of alkaline bromides and chlorides.* A. Cavazzi. Gazz. Chim. 13, 454.

FOR the separation of iodine from bromine and chlorine in combination with alkalis, a solution of neutral ferric sulphate to which 5 to 10 per cent. of ferrous sulphate is added, may with advantage replace ferric chloride. By boiling this solution with a mixture of the three haloid salts only iodine is liberated.—A. S.

## New Books.

TREATISE ON CHEMISTRY. VOL. III. ORGANIC CHEMISTRY, PART II. By H. E. ROSCOE, F.R.S., and C. SCHORLEMMER, F.R.S., Professors of Chemistry in the Victoria University, Owens College, Manchester. London: Macmillan and Co., 1884. 8vo. volume, bound in cloth, contains 630 pages of printed matter, and a full alphabetical index of subject matter, occupying nearly twenty pages.

THIS work forms Part II. of Vol. III. of the treatise on Organic Chemistry, and it commences with the consideration of the classes of organic compounds, containing diatomic or divalent alcohol radicals, and closes with that of the Tetrinic acid group, including Thiophene. To give an idea of the scope of this volume (Part II.), the following are the representatives of the various groups discussed in it: Compounds containing diatomic or divalent alcohol radicals. Dyad alcohol radicals. The methylene compounds. The ethylene compounds. The ethidene or ethylidene compounds. The glycolyl compounds. The oxalyl compounds. The propylene compounds. The trimethylene compounds. The dimethylene compounds. The propylene compounds. The lactyl compounds. The paralactyl compounds. The hydraeryl compounds. The pyruvyl compounds. The malenyl compounds. The butylene and trichlorbutidene and succinyl compounds. The pentylene, hexylene, heptylene, octylene and higher compounds, with their oxy-acids, etc. The uric acid derivatives. Compounds of trivalent or triad radicals. Compounds of the monad-alcohol radicals,  $\text{C}_n\text{H}_{2n-1}$ , and of the acid radicals,  $\text{C}_n\text{H}_{2n-3}\text{O}$ . The allyl compounds. The acryl and the crotyl compounds. Compounds containing from ten to fifteen atoms of carbon. Tribasic acids,  $\text{C}_n\text{H}_{2n-1}(\text{CO}_2\text{H})_3$ , and compounds related to these. Hydrocarbons of the acetylene series,  $\text{C}_n\text{H}_{2n-2}$ , and their derivatives. Alcohols of the series,  $\text{C}_n\text{H}_{2n-2}\text{O}$ . Acids of the series,  $\text{C}_n\text{H}_{2n-4}\text{O}_2$ . Drying oils. Alcohols of tetratomic radicals. Alcohols of hexatomic radi-



cals. Carbo-hydrates. The sugar group, the amylose group. The glucoses. The amyloses. The gums. Cellulose. The furfuryl group. The meconic acid group. The tetrinic acid group. The attention of technologists may be specially directed to the chapters on glycerine (glycerol) oxalic acid, starches, sugars and drying oils. Interspersed with the text are 49 excellent engravings, and numerous references to original sources are recorded below the text, thus adding greatly to the value of the work. A German edition of this work has also been simultaneously issued, the publishing firm being that of Friedrich Vieweg und Sohn, Braunschweig.

**WORKSHOP RECEIPTS FOR MANUFACTURERS, MECHANICS AND SCIENTIFIC AMATEURS.** (Third series). By C. G. WAINFORD LOCK. London: F. and F. N. Spon, 16, Charing Cross. New York: 35, Murray Street, 1884. Svo. volume containing frontispiece, preface, table of contents, and subject matter, covering 468 pages. An alphabetical index covering twelve pages closes the work. The matter is arranged in the similar alphabetical order observed in Dictionaries of Arts, etc., or Encyclopedias, and interspersed with it are 183 wood-cuts. Immediately preceding the index is an appendix, devoted to an article on Aluminium.

The frontispiece equally alluded to is an excellent chromolithographed colour scale for tempering iron. The following are the subjects treated of in the work: Alloys, aluminium, antimony, barium, beryllium, bismuth, cadmium, caesium, calcium, cerium, chromium, cobalt, copper, didymium, electrics, enamels and glazes, erbium, gallium, glass, gold, indium, iridium, iron and steel, lacquers and lacquering, lanthanum, lead, lithium, lubricants, magnesium, manganese, mercury, mica, molybdenum, nickel, niobium, osmium, palladium, platinum, potassium, rhodium, rubidium, ruthenium, selenium, silver, slag, sodium, strontium, tantalum, terbium, thallium, thorium, tin, titanium, tungsten, uranium, vanadium, yttrium, zinc, zirconium.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1884.

6455 E. A. Brydges, Berlin. Improvements in the process of manufacturing hydrocarbon gas and in apparatus therefor. Communicated by W. F. C. Mason McCarty, St. Petersburg, Russia. Complete specification. April 17.

6464 W. Virgo Wilson, London. Improvements in the manufacture of lead soap, especially for their application to the manufacture of white lead. April 17.

6472 C. D. Abel, London. An improvement in the manufacture of hard soap. Communicated Die Fabrik Chemischer Produkte (Aktien Gesellschaft), Berlin, Germany. April 17.

6484 John Giers, Middlesbrough, Yorkshire. Improvements in the manufacture of steel by the pneumatic or Bessemer process, and apparatus therefor. April 17.

6490 A. Rycie, Hebburn-on-Tyne, Durham. Improvements in the protection from fouling of iron, steel, and wooden ships' bottoms, to be called Rycie's Oceanic Composition. April 18.

6513 J. H. Johnson, London. Improvements in or connected with furnaces or heating apparatus for burning liquid hydrocarbons. Communicated by J. Buffet, Paris. April 18.

6514 T. Nordenfelt, Westminster, and V. A. Meurling, Christianstad, Sweden. Improvements in the manufacture of gunpowder and like explosive compounds. Complete specification. April 18.

6515 T. Nordenfelt, Westminster, and V. A. Meurling, Christianstad, Sweden. Improvements in the manufacture of gunpowder and other explosive compounds. April 18.

6517 F. Ransome, Lower Norwood, Surrey. Improvements in the manufacture of cement. April 18.

6520 E. K. Muspratt, Liverpool, and G. Eschellman, Ph. D., Widnes. Improvements in the manufacture of magnesia. April 18.

6528 E. F. Trachsell, London. An improved method or process of producing oxide of strontium, of barium, or of calcium. April 19.

6543 C. Espir, Wimbledon, Surrey. Improvements in the manufacture of powder for blasting and other analogous purposes. April 19.

6555 A. C. Henderson, London. Improvements in the method of, and in apparatus for manufacturing carburetted hydrogen gas for either lighting or heating purposes. Communicated by C. Wilhelm, Paris. April 19.

6556 E. Johnson, Newport, I. of Wight. Making anticlinker carbon crete fuel. April 19.

6557 G. Epstein, London. Improvements in the treatment of grain or other materials for the production of alcohol therefrom. April 19.

6584 B. J. B. Mills, London. Improvements in calcining furnaces. Communicated by H. S. Firman, New York, U.S.A. April 19.

6620 H. Sney, Roubaix, France. Malting the grain used in the fabrication of beer. April 21.

6627 W. R. Lake, London. An improved process for the manufacture of linseed oil and apparatus therefor. Communicated by H. A. Davidson, Buffalo, U.S.A. Complete specification. April 21.

6631 M. Schwab, Manchester. Improvements in apparatus for making illuminating gas. Communicated by J. Overhoff, Gummersbach, Germany. April 21.

6643 J. Clark, Nelson, Lancashire. An improved compound or composition to be used for sizing yarns. April 22.

6652 J. H. Johnson, London. Improvements in apparatus for carburetting air. Communicated by E. Delamere-Debouteville and L. P. C. Malandin, Fontaine-le-Bourg, France. February 26. (Previously included in No. 3986).

6659 G. S. A. Albright, Oldbury, Worcestershire. Improvements in self-igniting and inextinguishable signal lights for signalling at sea or on lakes and other waters. April 22.

6664 J. Walker, Leeds. The preparation or manufacture of drinks or beverages. April 22.

6666 A. W. Lovell Iddie, London. Improvements in kilns for drying or roasting malt. Communicated by F. Pelzer, Dortmund, Germany. April 22.

6699 P. Mackintyre, Maryport, Cumberland. The production and injection of hot gasses into the blast furnace, and apparatus therefor. April 23.

6700 E. Jackson, Handsworth, Staffordshire. An improved process for the extraction, concentration, and decolorization of glycerine, and for obtaining other useful products from the spent leys of soap making. April 23.

6707 E. Perrins, Sparkbrook, Warwickshire. Improvements in the manufacture of unfermented drinks. Complete specification. April 23.

6720 E. Capitaine, London. Apparatus for generating carbonic [sic], maintaining any required pressure. Communicated by Dr. Max Schroeder, Berlin. April 23.

6722 A. P. Price, London. Improvements in obtaining copper. April 23.

6724 G. Singer and C. D. Singer, London. Process or method of making paper-stuff out of esparto, wood, straw, and the like, and apparatus therefor. Communicated by J. H. Chaudet, Rouen, France. April 23.

6728 E. P. Alexander, London. Improvements in malting grain, and in the machinery, apparatus, or means to be employed therein. Communicated by J. N. Galland, Paris. April 23.

6731 W. J. A. Donald, Glasgow. Improvements in the manufacture of chromates and bichromates. March 13. (Previously included in No. 4311).

6736 Dr. C. Hoepfner, Berlin. Improvements in the process of decomposing metallic haloid salts by electrolysis. April 23.

6737 R. Tervet, Johnstone, J. Pettigrew, jun., Greenock, and N. MacPhie, Johnstone, Renfrewshire. Improvements in processes for treating sulphates to obtain carbonates, hydrates, or oxides, and easily utilisable by-products. April 24.

6742 J. Clare, Penketh, Lancashire. Improvements in packing caustic soda. April 24.

6750 E. Packard, jun., Ipswich, Suffolk. Improvements in manufacturing very pure and rich superphosphates of lime. April 24.

6751 E. Packard, jun., Ipswich. Improvements in obtaining from native or other phosphate of lime products rich in phosphoric acid and of a high degree of purity. April 24.

6752 E. Packard, jun., Ipswich. Improvements in obtaining from native or other phosphate of lime products rich in phosphoric acid. April 24.

6765 J. Guy Wilson, Manchester. Improvements in crucibles and other vessels for melting, refining, or converting metals, and means for heating the same. Communicated by J. E. Bott, Linwood, Delaware, U.S.A. April 24.

6768 J. Wetter, Westminster. Improvements in the method of and apparatus for making fodder from certain waste products. Communicated by H. Hencke & Co., Gröneck, Germany. April 24.

6779 E. Fischer, London. Improvements in the manufacture of oxidised linseed oil. April 24.

6786 W. P. Thompson, Liverpool. Improvements in the bleaching or purifying of dark-coloured mineral and other distillable oils. April 25.

6789 R. Robinson, Bishop Auckland, Durham. An apparatus for washing coal or other minerals. April 25.

6805 W. Scheidt, Kettwig-on-the-Ruhr, Prussia. A method of and apparatus for washing, drying, bleaching, rinsing, impregnating, and drying fibrous materials. Complete specification. April 25.

6808 J. H. Porter and J. Porter, London. Improvements in filter presses. April 25.

6831 H. G. Williams East Greenwich, Kent. An improved apparatus for amalgamating auriferous, argentiferous, or other metalliferous ores or substances. April 25.

6861 J. Wetter, London. New or improved processes for producing colouring matters from phenol. Communicated by The Leipziger Anilinfabrik Beyer and Kegel, Lindenau, Germany. April 26.

6872 W. R. Lake, London. Improvements in and relating to the manufacture of colouring matters. Communicated by L. Vignon & Co., Lyons, France. April 26.



- 6901 N. A. Helouis, Paris. An improved process of manufacturing light essences by the distillation of coal, wood, turf, oil, and other similar substances. April 28
- 6931 D. Stewart, Glasgow. Improvements in centrifugal apparatus for drying sugar or other granular matters. Complete specification. April 29
- 6936 E. Metcalf, Accrington, Lancashire. Preserving and preventing incrustation in steam boilers, which he names "The new anti corrosive preparation." April 29
- 6937 W. P. Thompson, Liverpool. An improved manufacture of oil, applicable for use in railway lamps, and for other purposes. Communicated by J. Scrafton, Lahore, India. April 29
- 6968 A. W. L. Reddie, London. Improvements in furnaces for burning green sugar-cane and similar moist substances. Communicated by M. Agrela, Paris. April 29
- 6973 F. W. Renaut, Burstow, Surrey. A ferment for use in indigo vats, and in vats for dyeing with noir bleu, or with mixtures of noir bleu and indigo. Communicated by C. Collin and L. Benoist, Paris. April 29
- 6974 F. W. Renaut, Burstow, Surrey. Improvements in indigo vats, and in vats for dyeing with noir bleu or with mixtures of noir bleu and indigo. Communicated by C. Collin and L. Benoist, Paris. April 29
- 6982 A. M. Clark, London. Improvements in apparatus for purifying and hydrating gases. Communicated by W. Maynard, New York, U.S.A. April 29
- 6983 A. P. Price, London. Improvements in the purification of coal gas, or of other gases containing ammonia or ammoniacal compounds. April 29
- 6985 B. J. B. Mills, London. Improved apparatus for carbonizing bones and turf, and for coking and distilling brown-coal, pit-coal, and other carbonaceous materials by means of superheated steam. Communicated by A. Zwillinger, Vienna. April 29
- 7019 A. Maitland, Pollockshields, Renfrewshire. Improvements in the manufacture of barm. April 30
- 7054 J. N. Soderholm, London. Improvements in lucifer matches. April 30
- 7056 Sir P. C. Knowles Lovell Hill, Bart., Ryde, Isle of Wight. A new or improved method of treating sulphides and oxides of metals and obtaining products therefrom. Jan. 3. (Previously included in No. 603.)
- 7059 A. Frank, Ph.D., Charlottenberg, Prussia. Improvements in the treatment of slag and other phosphates containing sulphur and phosphorus. April 30
- 7097 J. Wetter, Westminster. A process or processes for producing betanaphthol-sulpho-acids, separating the same, and obtaining colouring matters therefrom. Communicated by The Leipziger Anilinfabrik, Beyer and Kegel, Lindenau-Leipzig, Saxony. May 1
- 7098 J. Wetter, Westminster. A process or processes for separating the different betanaphthol-monosulpho-acids from a mixture of the same, and for obtaining colouring matters therefrom. Communicated by The Leipziger Anilinfabrik, Beyer and Kegel. May 1
- 7107 J. Inray, London. Process and apparatus for the manufacture of soda by the ammonia treatment. Communicated by La Société Anonyme des Produits Chimiques du Sud-Ouest, Paris. Complete specification. May 1
- 7108 H. H. Lake, London. Improvements in presses or apparatus for the manufacture of compressed gunpowder. Communicated by H. Gruson, Buckau-Magdeburg, Germany. May 1
- 7111 J. Watson and J. Lamb Spoor, Gateshead-on-Tyne. Improvements in and connected with kilns employed in the manufacture of Portland cement. May 1
- 7145 J. H. Johnson, London. Improvements in or connected with the covering of surfaces with decorative or protective coatings or films. Communicated by A. Semal, Nivelles, Belgium. May 2
- 7156 D. Tildesley, Brondesbury, Middlesex. Improvements in apparatus for injecting fuel and hot air into furnaces, for injecting liquid and solid matter into retorts for gas-making, and for aerating condensed water. May 2
- 7158 A. Grothe, London. Improvements in the process of and apparatus for burning pyrites. May 2
- 7175 A. Neilson, Inkermaun, Renfrewshire. Improvements in retorts or apparatus for distilling shale or other oil-yielding minerals. May 3
- 7176 J. A. Yeadon, Leeds. Improvements in the drying of coal, coke, or other similar materials. May 3
- 7198 S. K. Page, Aylesbury, C. E. Robinson, Scarborough, and W. Stevens, London. Improvements in apparatus for use in the treatment of sewage, and in other operations in which solid matter is required to be added to or mixed with fluid matter in certain relative proportions. May 3
- 7199 J. Jeyes, Plaistow, Essex. Improvements in the manufacture of elastic compounds. May 3
- 7200 J. Kirkaldy, London. Improvements in surface condensers and heaters. Complete specification. May 3
- 7206 J. Hargreaves, Widnes, and T. Robinson, Farnworth. Improvements in treating ores and substances containing antimony to obtain metallic antimony and other products, and in appliances employed therein. May 5
- 7223 R. C. Jay, Bayswater. An improved apparatus for blowing, forcing, or exhausting air, gas, or vapour, and for charging them with the vapour or gas of oils, liquids, or fluids. May 5
- 7230 J. E. Billups, Cardiff. The manufacture of hydraulic cement from the calcining and mixing of the limestone pebbles found in the Bristol Channel (usually termed Aberthaw pebbles), or the limestone rock from which the same are formed, and Bristol Channel mud or clay, or the clay found between the layers of the said lime stock and silica. May 5
- 7235 V. Daix, St. Quentin, France, and A. L. Possoz, Paris. Improvements in the extraction of sugar from molasses and other saccharine products, and in the quality of alkaline salts resulting therefrom. May 5
- 7256 G. King, Beckton, Essex. Improvements in fastenings for retort lids. May 5
- 7257 J. Beasley, Handsworth, Staffordshire. Improvements in the manufacture of iron and in the furnace linings to be used in said manufacture. May 5
- 7255 M. Williams, Wigan. Improvements in the method of preparing lime for sanitary purposes. May 5
- 7258 E. Solvay, Brussels. Improvements in the manufacture of chlorine. May 6
- 7259 E. Solvay, Brussels. Improvements in the manufacture of chlorine and chloride of lime. May 6
- 7260 E. Solvay, Brussels. Improvements in the manufacture of hydrochloric acid. May 6
- 7304 D. H. Brandon, London. An improved asbestos compound for use as boards or sheathing for roofings, wearing surfaces of car brakes, and for building and other purposes. Communicated by D. A. Brown, Boston, U.S.A., and C. F. Brigham, Worcester, U.S.A. Complete specification. May 6
- 7305 D. H. Brandon, London. An improved fire and water-proof material for roofing, sheathing, and other purposes. Communicated by D. A. Brown and C. F. Brigham, as above. Complete specification. May 6
- 7328 J. Inray, London. An improvement in the manganese treatment of iron for the production of steel. Communicated by the Société des Acieries de Longwy, Longwy, France. May 6
- 7342 C. Troop, Stepney. Improvements in the manufacture of fireproof blocks or slabs, and in means of protecting the joints of buildings from fire. Complete specification. May 6
- 7353 O. Roberts, Liverpool. Improvements in scrubbing apparatus for purifying gas. May 7
- 7357 J. Forsyth Johnstone, Bow Common, and J. Bingham Elliott, London. Improvements in apparatus for drying waste animal matters, fish, and other materials, applicable also to the concentration of liquids. May 7
- 7388 J. F. C. Farquhar, London. Improvements in apparatus for filtering liquids. May 7
- 7395 and 7397 J. A. Jones, Middlesbrough-on-Tees. Improvements in the manufacture of concrete or artificial stone for paving or other purposes. May 8
- 7403 W. P. Thompson, Liverpool. Improvements in the utilization of slags containing phosphoric acid produced by the basic process for desphosphorizing pig iron. Communicated by R. Solihwa, Dortmund, Germany. May 8
- 7404 W. P. Thompson, Liverpool. Improvements in the purification of iron ores and of iron by the basic process. Communicated by R. Solihwa, Dortmund, Germany. May 8
- 7405 G. Kemp, Swinton, Yorkshire. Improvements in glass melting tanks. May 8
- 7413 J. Rust, Clapham, Surrey. Vitreous cement. May 8
- 7418 G. H. Anderton and W. W. Mellon, Howden, Yorkshire. The production of soluble phosphates of lime, rich in phosphoric acid. May 8
- 7425 J. H. Johnson, London. An improved washing soap. Communicated by E. Bar, Paris. May 8
- 7433 A. Schröder, Dresden, Germany. Improved apparatus for the manufacture of nitro-glycerine. May 8
- 7438 J. Chabanel and A. V. Brabant, care of A. W. Clark, London. Improved apparatus for disinfecting, separating, and filtering sewage matters. May 8
- 7448 A. French, North Shields. An improved process for making a black pigment and polishing material. May 9
- 7458 J. Clare, Penketh, Lancashire. Improvements in packing bleaching powder. May 9
- 7478 M. Heddicke, London. Utilization of hops in the manufacture of intoxicating beverages, otherwise than beer. May 9
- 7481 C. Waite, Reading, Berkshire. Filtering of sewage by precipitation; utilizing the deposit, and rendering harmless the effluent water. May 9
- 7486 A. B. Reck, Copenhagen. A brick-lined hot-air furnace. May 9
- 7487 A. B. Reck, Copenhagen. An iron hot-air furnace. May 9
- 7496 A. M. Clark, London. A new or improved compound for preventing the formation of clinkers in furnaces. Communicated by The Coal Economizing Company, Topeka, Kansas, U.S.A. May 9
- 7501 S. De La Grange Williams, Birmingham. Improvements in kilns for burning lime and cement, and for other like purposes. May 9
- 7502 L. Benelmans, H. Tilmans, and E. Marlier, Brussels. A new process for condensing acid gas and vapours, and for manufacturing iron salts. May 9
- 7510 A. Ponsard, Paris. Improved means and apparatus for the manufacture of gas for lighting purposes and for heating purposes. May 9
- 7511 J. H. Johnson, London. Improvements in bleaching paper pulp or other materials. Communicated by J. B. Jessy, St. Etienne, France. May 9
- 7525 C. Wigg, Liverpool. Improvements in the manufacture of carbonate of soda. May 10
- 7526 W. A. Meadows, Rainhill, Lancashire. Improvements in the manufacture of sulphate of ammonia. May 10
- 7537 J. F. Scott, Withington, Lancashire. Improvement in the carbonization or destructive distillation of wood, and in the treatment of the gases therefrom. May 10
- 7540 G. Dalton, Leeds. Improvements in machines for crushing stones, ores, and other hard and brittle substances. Complete specification. May 10
- 7562 F. W. Harbord, Wolverhampton. Improvements in the manufacture of phosphates. May 10



7566 A. Pensard, Paris. Improvements in the manufacture of homogeneous iron, of steel, and of other cast metals, and apparatus therefor. May 10

7569 E. F. Trachsel, London. An improved method or process of treating sulphates and sulphides for obtaining oxides or carbonates and recovering bye-products therefrom. May 10

7570 E. F. Trachsel, London. An improved method or process of producing oxides or carbonates from chlorides, and recovering bye-products therefrom. May 10

7573 J. W. Freestone, Bromborough, Cheshire. Improvements in separating glycerine from animal and vegetable substances. May 10

7588 W. Whittaker, Burnley, Lancashire. Improvements in furnaces for the combustion of animal, vegetable, or town's refuse. May 12

7615 I. Levinstein, Manchester. Improvements in preparing or treating and utilizing certain waste substances. May 13

7619 J. Crighton, Manchester, J. Walsh, Middleborough-on-Tees, and R. Crighton, Manchester. Improvements in the manufacture of crude oxalate, and in the construction of apparatus used in such manufacture. May 13

7640 W. Beardmore, and J. MacCallum Cherrie, Glasgow. An improvement in the manufacture of steel. May 13

7645 H. J. Haddan, London. Improvements in the manufacture of violet colouring matters. Communicated by Dr. H. Hassenkamp and the Farbenfabriken, vorm. F. Bayer and Company, Elberfeld, Prussia. May 13

7646 H. J. Haddan, London. Process or processes for the production of soluble combinations of certain azo compounds with bisulphites, and for utilizing the same in dyeing and printing. Communicated by Dr. H. Hassenkamp and the Farbenfabriken vorm. F. Bayer and Company. May 13

7651 and 7655 P. M. Justice, London. An improved process of treating cotton seed. Communicated by The United States Cotton Seed Cleaning Company, New York. May 13

7693 J. Swift, London. Ether freezing micrometer. May 11

7694 H. A. Costerton, Brighton. Improvements in gas carbonators, or apparatus for introducing into ordinary gas the vapour of a volatile liquid carbon; with the object of enriching the said gas for illuminating or heating purposes. May 11

7707 H. J. Haddan, London. Improvements in the manufacture of castings from furnace slag. Communicated by L. Perin, St. Amand, France. May 11

7712 E. Elsasser, Barmen, Prussia. Improvements in the manufacture of several monosulpho-acids of betanaphthylamine. May 11

7713 E. Elsasser, Barmen, Prussia. Improvements in the manufacture of red azo dyestuffs or colouring matters. May 11

7731 E. H. Cook, Bristol. Preventing the emission of metallic vapours, compounds of metal, sulphur dioxide, or other vapours from the chimneys and flues of chemical, metallurgical, or other works. May 15

7772 W. E. Brendon, Exeter. Improvements in apparatus or appliances for precipitating copper from liquids containing copper in solution. May 15

7783 C. Price, Leicester. A novel and improved air blast or exhaust kiln, with one or more chambers for burning bricks, pipes, tiles, or earthenware articles, whereby a great saving in time and expenses can be gained. May 16

7810 W. H. Wills, Liverpool. An apparatus for cooling brewers' and distillers' worts, and any liquid, and the condensing of steam. May 16

7811 A. M. Clark, London. Improvements in the manufacture of sodic, potassic, and other sulphhydrates, and in their application in the manufacture of cellulose pulp for paper. Communicated by R. Blitz, Paris. May 17

7817 F. W. Renaut, Burstow, Surrey. Improvements in obtaining from alkali-waste sulphuretted hydrogen and calcium carbonate. Communicated by Dr. H. von Miller, Hruschau, Austria. Complete specification. May 17

7850 E. Page and R. Itanger, Maidstone. The equalisation of the specific gravity of worts in the manufacture of beer. May 17

7890 J. Crutchett, New York, U.S.A. Mode of improvement for the manufacture, cleansing, and condensing of steam carbon-gas for the uses of heat, light and power. May 17

7893 W. Clarke and H. J. Tansley, Nottingham. Improvement in dyeing mixed cotton and silk, lace, and other fabrics. May 17

7871 D. Urquhart, Westminster. Process for removing sulphates and obtaining sulphuretted hydrogen from potash lyes. May 17

7878 D. L. Collins, Grays, Essex. Improvements in kilns for burning Portland cement. May 17

7884 S. Delbarre, London. Improvements in oxygenating water. Communicated by A. Brin, Paris. May 17

7892 A. H. Cochrane, Dublin. An effervescing and still non-alcoholic malt beverage. May 17

7900 W. E. Gedge, London. Improvements in apparatus and process for boiling, soaking, bleaching, dyeing, washing, and drying fibrous materials. Communicated by F. C. Glaser, Berlin. May 19

7909 W. Mather, Manchester. Improvements in bleaching, and in apparatus for the purpose. May 19

7914 H. H. Minns, Maidenhead. Improved method and apparatus for rendering paper, leather paper, and other materials damp-proof, the apparatus being also applicable for pasting or cementing various thicknesses of material together. May 19

7922 G. J. Heathern, Weymouth. Improvements in the purification of musty beer. May 19

7933 A. M. Clark, London. Improvements in tanning hides and skins. Communicated by W. Maynard, New York, U.S.A. Complete specification. May 19

7940 A. M. Clark, London. A new or improved process of treating iron. Communicated by R. B. Abbott, Freeborn, Minnesota, U.S.A. May 19

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## NOTICES.

### ANNUAL GENERAL MEETING.

The next Annual General Meeting of the Members of the Society of Chemical Industry will be held at Newcastle-on-Tyne, on Wednesday, Thursday, and Friday, the 9th, 10th, and 11th of July next.

On the morning of Wednesday, July the 9th, at eleven o'clock, the Members will meet in the Lecture Theatre of the Literary and Philosophical Society, Westgate Road, to transact the ordinary business of the Society, to elect Office Bearers, and to hear an address by the President. In the afternoon they will visit the works of Sir W. G. Armstrong, Mitchell, and Company, at Elswick; and in the evening the Annual Dinner will be held in the Banqueting Hall, Jesmond Dene, by the kind permission of Sir W. G. Armstrong, C.B. Dinner tickets, 10s. 6d. each, exclusive of wine, may be obtained on application, accompanied by Cash or Money Order, to the Honorary Secretary of the Newcastle Section, Mr. J. T. Dunn, 29, Oxford Street, Newcastle-on-Tyne.

A change has been made in the proposed arrangements for the second day of the meeting. It is now arranged that on the morning of Thursday, July the 10th, the Members will proceed by special train to the Lead Works of Messrs. Locke, Blackett, and Company, at St. Anthony's; then to the Alkali Works of the Newcastle Chemical Works Company, at Gateshead; and in the afternoon to the Works of Palmer's Shipbuilding and Iron Company. In the evening a *Conversazione* will be held in the Assembly Rooms, Westgate Road. Tickets for the *Conversazione* will be obtainable at the Reception Room during Wednesday and Thursday.

On Friday, the Members will meet at the Swing Bridge, after inspection of which they will proceed by steamer down the river to Hebburn, where the Alkali Works of Messrs. Charles Tennant and Company, and the Metal Extracting Works of the Tharsis Sulphur and Copper Company, will be visited. After luncheon in the Drill Hall of 1st Newcastle Volunteer Artillery, the steamer will take the Members across to Willington, to inspect the Lead Works of Messrs. Cookson and Company. The Members will then proceed by the steamer down the river to the pier at Tynemouth, then returning to Newcastle either by steamer or by train, as they may prefer.

The Wood Memorial Hall of the North of England Mining Institute will be available during the meeting as a Reception and Writing Room. The Library of the Literary and Philosophical Society will also be available as a Writing Room.

Various Works in the district, in addition to those mentioned above, will be open to Members during the Meeting, on presentation of their cards of membership.



Opportunities of seeing in operation the Simon-Carvès process, and the Jameson process, will be afforded during the Meeting.

Messrs. Baird and Company have consented that any Members attending the Meeting who choose to proceed to Gartsherrie for the purpose, shall see the process at work there for obtaining ammonia from the gases from blast furnaces; and Messrs. Young and Beilby have promised to show their process at two Works near Edinburgh.

A sketch programme of the arrangements for the Meeting is issued separately with the present number of the Journal. A complete programme, with map and plans of Newcastle and district, together with Hotel, Railway, Conveyance, and Postal information, will be furnished to Members at the Reception Room.

An early application for Dinner Tickets is especially desired.

Numerous inquiries have been made for sets of the Journal for 1882. Some of the numbers for that year are out of print. Members who have not the volume for 1882, and wish to obtain it, are invited to signify that wish to the Secretary as early as possible. The numbers out of print are now being reprinted, and will be ready shortly.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 36, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

#### LIST OF MEMBERS ELECTED JUNE 23rd, 1884.

G. Mander Allender, Solna, Rochampton, S.W., farmer.  
Thos. Bennett, Birch Vale, near Stockport, calico printer.  
Thos. Bevan, 122, Cheshire View, Broad Street, Pendleton, manager of print works.  
Thos. Bolas, 8, Grove Terrace, Chiswick, W., consulting chemist.  
Patrick Carmody, Laboratory, Somerset House, W.C., analyst.  
G. H. France, Horsforth, Leeds, dyer.  
Andrew French, 29, Washington Terrace, North Shields, metallurgist.  
H. D. Hardie, 30, Woodlands Road, Glasgow, drysalter.  
R. R. Kelly, 3, Pall Mall East, London, S.W., chem. manuf.  
Dr. Ludwig Landsberg, Normal School of Science, South Kensington, S.W.  
Wm. J. Leonard, Hope Chemical Works, Hackney Wick, E., naphtha distiller.  
Frank Livesey, South Metropolitan Gas Co., 709A, Old Kent Road, S.E., engineer.  
Thos. Macfarlane, 16, Inspector Street, Montreal, Canada, chemist (temporary address) c/o Lister and Biggs, 3, Laurence Pountney Hill, E.C.  
R. W. McKinlay, Ameliaville, Aytoun Road, Pollokshields, N.B., metal merchant.  
D. Melville, Chemiske Fabrik, Stavanger, Norway, manager.  
G. Rawlins, Brook Works, Rainhill, Lancashire, ultramarine maker.  
J. W. Robinson, Junr., 3, Gladstone Terrace, Gateshead, merchant.

R. Robinson, Green Street, Tonge, Middleton, Lancashire, dyer.  
W. Cameron Sillar, St. James's Lodge, Kidbrook Park Road, Blackheath, S.E., director of Native Guano Co.  
Jos. Singleton, 41, Corporation Street, Manchester, drysalter.  
Wm. Stevens, 29, New Bridge Street, Blackfriars, E.C., secretary.  
James Tennant, Dartmouth Lodge, Saltwell, Gateshead, alkali maker.  
T. Thompson, Skeldergate Bridge, York, chem. manuf.  
Jos. Verdin, The Brockhurst, Northwich, Cheshire, salt proprietor.

#### CHANGES OF ADDRESS.

W. Dalrymple Borland (late of Stoke Newington), The Explosives Co. Limited, Stowmarket, Suffolk.  
Geo. E. Davis (late of Fallowfield), Belmont, Thorncliffe, Sheffield.  
Archibald Duncan (late of Renfrew), 1, Teneriffe Street, Higher Broughton, Manchester.  
Thos. Good (late of Somerset House), Inland Revenue, Alfreton, Derbyshire.  
T. G. Greenway, 28, Wilkinson Street, Sheffield (not Brownswood Park).  
Thos. Griffiths (late of Birkenhead), 10, King's Road, Clapham, S.W.  
A. H. Mason (late of Jean Baptiste Street), 178, Mance Street, Montreal.  
Robert Mullard (late of Bermondsey), 46, Drakefell Road, St. Catherine's Park, Hatcham, S.E.  
T. H. Muras (late of Runcorn), 33, Lovaine Crescent, Newcastle-on-Tyne.  
F. G. Roberts (late of Upper Clapton), Clydach, Bethune Road, Stamford Hill, N.  
H. Simon (late of St. Peter's Square), 20, Mount Street, Manchester.  
Geo. Valentine (late of Pumpfields), 11, Tatham Street, Liverpool.

#### CHANGES OF ADDRESS REQUIRED.

G. Chaloner, late of 247, Essex Road, N.  
F. G. Holmes, late of 50, Moneriff Street, Peckham, S.E.  
W. B. Symo, late c/o Jas. Guthrie, North Bridge Street, Bathgate, N.B.

### Deaths.

MRS. U. J. GROSJEAN, 97, Euston Road, N.W.  
E. JACKSON, 59, Boulton Road, Handsworth, Birmingham.

#### NOTICE.

The General Secretary will leave for Newcastle on Monday, the 7th July. All communications from that date until the end of the Meeting should be addressed to him, care of J. T. Dunn, Esq., Oxford Street, Newcastle-on-Tyne.

### London Section.

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Local Sec.: Thos. Tyrer, Garden Wharf, Church Road, Battersea, W.

The publication of the Paper on "The Processes concerned in the Conversion of Starch into Alcohol, and their relation to Brewing and Distilling," by W. S. Squire, Ph.D., is postponed till July.

## Liverpool Section.

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Hon. Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be made to Edward George Ballard, Queen's Park, St. Helens.

UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL.

Wednesday, April 2nd, 1884.

PROFESSOR J. CAMPBELL BROWN IN THE CHAIR.

## ON METHODS OF SAMPLING.

BY A. NORMAN TATE, F.I.C.

I WAS unable to attend the meeting of this section held last November, when a discussion took place on "International methods of sampling and testing Commercial products"; but in a letter addressed to the Chairman, with reference to the circular of Dr. Lunge, I ventured to express the opinion that an International agreement as to the modes of sampling and testing might be arrived at.

In discussing this question, I consider that we should first, fully and fairly inquire into all the several principles involved in sampling operations; for certainly in the operation of assessing the value of Commercial products, sampling is most important, for if the sample does not truly represent the bulk all further work is valueless. I believe that when all details of the characters of the substances to be sampled, nature of packages, changes which the substance may undergo after packing, and the conditions under which sampling may take place, are properly considered, satisfactory methods of sampling can be defined.

Dr. Hurter remarked, when discussing the subject at the meeting referred to, "Sampling will probably have to be left to the intelligence of the sampler. Samples have to be drawn under so many difficult circumstances as to localities, accessibility, and packages, that a rigid rule could not be drawn up, which can be enforced at all times, and under all conditions." No person conversant with sampling would expect this, but it may reasonably be hoped that certain general rules, based upon the character of the substance sampled, and the conditions surrounding it under different circumstances, may be drawn up and acted on by persons specially trained as samplers. The difficulty now, so far as I see, lies in the fact that samples are drawn too often by persons without any knowledge or appreciation of the characters of the substances to be dealt with.

It may be well to mention some few well-known substances, and the conditions surrounding their sampling, so as to point out some leading principles which, in my opinion, should be attended to. When such substances are sampled, there are certain items leading to discrepancies which are of the same general character, as for example the presence of moisture, the proportion of which is too frequently overlooked. Other items have special reference to special substances. One preliminary that has to be considered is the time and place of sampling, so that if a special strength or quality is to be delivered, the place of delivery and mode of sampling there, shall be clearly

understood. As a matter of practical detail, this is not always to be accomplished, and oftentimes it happens that what might have been quite up to strength or guaranteed quality at point of delivery, may fall short at some later date.

With reference to the statement that samples should always be drawn by persons acquainted with the properties of the substances sampled there comes the question "Who is best qualified to draw samples?" I was surprised to find this question included in a discussion marked out a year or two ago for the members of the Institute of Chemistry, viz.:—"Should chemists sample?" As my answer to such an inquiry I would say that if a chemist desires his work to be really useful, he should take every possible precaution to ensure that the sample he tests represents what it is supposed to represent, and consequently should make himself thoroughly familiar with every minute detail that has to be considered before the sample can be properly drawn. At the meeting of the Institute of Chemistry, at which this subject was discussed, I was astonished to find some analysts propound the view that the chemist has simply to analyse the sample given him, and need take no note of the circumstances of its sampling. To such a view I strongly dissent, and would say, that it is essential that the chemist who makes the test should not only be able to analyse and devise methods of correct sampling, but if necessary be able to sample himself. This matter of preliminary sampling goes beyond the actual sampling from packages, having reference to the treatment samples may undergo before and after they reach the laboratory, until they are actually dealt with by the analyst. I fear that many well drawn samples are much altered in character in consequence of faulty treatment either in manner of mixing, averaging, or general treatment before the analytical process is commenced.

Referring to details that affect samples generally, I would mention as an example moisture. The fact that the amount of moisture existing at the time of sampling and at the time when the sample is presented for analysis may vary, is too frequently overlooked. Moist samples are not unfrequently presented to the analyst in paper parcels, or under other conditions which certainly must give rise to changes in the percentage of moisture. Amongst such samples are sugars, with the exception of beet-root, the samples of which are usually placed in well-sealed bottles. Samples of other sugars come to me in paper parcels with no protection against loss of moisture; so also sulphate of ammonia, nitrates of potash and soda, manures, many minerals, solid oils and fats, and in all these the proportion of moisture is an item of commercial account. I might add to this list considerably, but these substances will serve to show to practical men the fault I wish to point out.

Now as to the remedy. It would be no great difficulty, for any Committee, or other appointed body, to define that substances containing moisture should be so sampled that the amount they contain at time of sampling, may be correctly determined in course of analysis. Rules dealing with moisture should refer to sampling in wet weather, transit of samples uncovered or left in a damp atmosphere, and the placing in bottles or other vessels that can be securely fastened and made waterproof, and other items will suggest themselves to every practical man.

Again, whilst the substances which I have just alluded to lose, others may gain moisture, as for example, caustic soda, and caustic soda ash. These also should have their peculiarities carefully noted, and be dealt with accordingly. I would allude here to other changes that may take place owing to the hygroscopic properties of some substances. Much alteration may



occur in the outer layer of the sample as compared with the bulk, as for instance with caustic soda ash, and bleaching powder, whilst other substances which are not hygroscopic, may in wet weather become damp at points close to sides of package. In such cases the sampler should so sample that the sample is drawn from the entire bulk and not simply from the outer layer. One statement of a blunder made by a chemist who was called upon to sample, well serves to show what I mean. It had been arranged that a parcel of soda-ash should be sampled and examined by him. He proceeded to the works, and had the heads of two or three casks taken out; scraped off some of the immediately underlying ash, placed it in a bottle, took it away with him, and sent in his report. I need scarcely say that the parties to the dispute did not settle the matter on the basis of his report when the circumstances were made known to them.

Changes in transit owing to absorption of moisture from the atmosphere and other changes, cannot be eliminated from any discussion on proper sampling.

Again the physical condition of the sample must be noted. Are all the particles of the same size? If not, how far is it possible by proper riddling and weighing to note the relative proportions of lumps and smalls? In the case of many minerals, unless this is attended to, no proper average of the bulk can be reached. The same holds good with small lumps or coarse powder, and fine powder. I have in my hand a sample, contained, as you will see, in a small homœopathic tube, which was sent me by a chemist, who professes to examine commercial samples. This represents some 50 casks of caustic soda ash of rough character, with many large and small lumps and much fine powder. This so-called sample is not even powdered, with the object of getting something like an average, and if it had been most likely it would not have represented the bulk, and to bring such a sample before anyone as the representative sample of 50 casks of soda ash is an instance of either ignorance or gross neglect of circumstances pertaining to proper sampling. An amusing instance of what I consider must have been faulty sampling, took place in the well known Birkenhead coffee-chicory case, when several members of the Town Council met together and weighed out and mixed quantities of chicory and coffee, and after mixing in their way, sent samples to different analysts, and chuckled over the reports received as showing how widely analysts differ. It is a matter exceedingly difficult, unless special precautions are taken, to ensure a thorough average mixture of ground coffee and chicory. And although it may have given these inexperienced Birkenhead town councillors some pleasure in chuckling over the results, the reason of the differences would be clearly enough apparent to those thoroughly experienced in the sampling and mixing of such substances.

There are other points connected with sampling based upon the mode of division. For example, it is not unfrequently the case that one or two pieces of barks selected at random are handed in as samples of many packages to be tested for quinine or other alkaloids. Such a mode of sampling is grossly faulty. Seeds will often afford better opportunity for taking samples from different parts of bulk so as to get a fair average, but owing to different sizes of seeds, and many other characteristics, really good samples are rarely obtained unless special precautions are observed. A case was before me not long since, when many particles of sand, gravel, and dirt of various kinds, were mixed with bones (the bones of course being the valuable substance), but not the slightest care was traceable in the selection of the sample, for after it had been drawn from bulk and divided into separate parcels, one parcel contained

bones fairly free from gravel, sand, etc., and another containing almost the whole of such matters that had been drawn from the packages. The chemist's aid was invoked to determine the amount of siliceous matter in the different samples, and any person conversant with the details of such examination will readily see how widely the results must have differed.

I might add many other examples of faulty sampling as conducted by commercial brokers and merchants; in fact the process of sampling as conducted usually in mercantile offices requires very careful investigation and revision.

There is another cause of error in sampling, and that is the tendency of the bulk to separate into layers, when solid matters in suspension in liquids subside, and are not fully mixed up before a sample is taken from bulk. No doubt many examples of such kind will suggest themselves to practical men. I have found this often in the case of liquid extracts used for tanning purposes. These without experience might be supposed to be of the same character throughout the contents of a cask, without any tendency to separate into layers, but, oftentimes there is a thick dense layer at the lower part of the cask, and a more fluid layer at the top. This has, I know, given rise to many discrepancies in the tests reported, respecting the so-called *same samples* by different chemists. These same extracts, if carefully watched during different methods of sampling, will afford good examples of changes in percentage of the more valuable constituent. Another example of layers is shown with palm oil. When packed in Africa it is sufficiently liquid to allow of solid impurities and water settling out before the oil becomes quite solid. It therefore follows that every special precaution should be taken to note under what circumstances, and in what parts of the cask dirt and water may be found, as it is usual, and of course desirable, for a deduction from the total weight of the contents of the cask to be made, so that only oil is charged for.

Certain circumstances connected with a process of manufacture may often lead to strange results in sampling, and frequently through carelessness. For instance in refining oil, the oil may be placed in the tank, from which it is packed, before the liquid or chemical with which it has been washed or treated has subsided. In such cases, whilst the greater number of the casks packed may contain a thoroughly good merchantable product, one or two casks may have a large admixture of water and other impurities.

Such facts as these, and others met with by practical samplers, suggest the question, "From how many packages should a sample be drawn?" That is, from the whole number, or if not, from what proportion of the whole number. In many cases it is desirable that the contents of every single package should be alike, in other cases this is almost impracticable, and is not absolutely essential, provided the variation is not great. On this point much may be said, and all interested in correct sampling will do well to give the matter most careful consideration. Circumstances surrounding the sampling of commercial products often prevent the sample from being drawn from the whole of the packages, and, therefore, unless there is great uniformity in the strength and character of the contents of each, a really good average is not attainable. At the same time it may be fairly said, that no one package of any particular lot of goods, should vary widely from others. Persons accustomed to sampling on board ships, on flats, in railway trucks, in warehouses, and other places, will, I think, fully appreciate the difficulties I have just alluded to.

There are many other points I might mention with examples, concerning the sampling of very many other raw materials and manufactured products that I have

not alluded to. What I have said will perhaps serve to show the many difficulties that lie in the way of bringing about international or other agreements as to modes of sampling. Still, I think the attempt desirable, and believe the result attainable. But the matter must be dealt with only by those who are thoroughly familiar with all the details that have to be considered. A committee may be appointed, as has been suggested, of members of the Institute of Chemistry, Society of Chemical Industry, and Society of Public Analysts, to take preliminary steps and settle the basis of general work. Such committee could probably deal with such a question as the mode of taking and treating samples that contain moisture, and supply some course of procedure that would prevent many other disadvantages which now accrue from a disregard of necessary precautions, but a general committee would, I consider, be quite unable to deal with many matters of detail. Only such persons as are thoroughly familiar with every single detail pertaining to special articles can fully and fairly discuss methods of dealing with them. The general committee could make inquiry, and select persons having the confidence of certain trades, to deal with matters and products used and made by them, and these persons, whether members of the general committee or not, could report the results of their deliberations, which could then be forwarded to all persons interested, and, if necessary, meetings of representatives of such trades could be called to discuss the question more fully. There already exist different trades associations which could be invited to discuss these matters, and bring to bear upon them special knowledge in matters of sampling. Special knowledge of all minute details is absolutely essential, and the introduction of theoretical considerations only, without the test of practice, may become a source of error.

You will see from what I have said that my suggestion is a general committee composed of members of different societies interested in the taking of correct samples. Such a committee to deal with general principles, to watch for instances of incorrect procedure, and take steps to appoint proper persons to consider each special subject, arrange for the circulation of experts' report to the trades and persons interested, and adopt methods to get decisions arrived at, and made practically serviceable as rules for guidance under the special circumstances of each case. I have already alluded to what I consider an important element connected with sampling, viz., that chemists should give more attention than many do to methods of sampling, and study in every possible way the several characteristics of the substances they have to examine, so as to be able not only to indicate how samples should be drawn, but, if necessary, be capable of superintending the operation or performing it themselves. I think this cannot be urged too strongly. Then again it is, I think, desirable that more care should be taken in training men for the special work of sampling. There are already many men engaged in different seaports and manufacturing centres who have had lengthened and varied experience as samplers, and who know the behaviour of the substances they deal with under almost every conceivable set of circumstances. The aid of these experienced men is frequently valuable. Many of the men engaged in the London docks and wharves are fine examples of what can be done by training in sampling, and I have often noted with pleasure the knowledge of detail and care in manipulation which they bring to bear on the sampling of different substances. I must not omit, in conclusion, to mention care on the part of the chemist in the putting aside, under proper circumstances and conditions, of samples that he has

examined, so that they may undergo no change, and be obtainable for reference should disputes arise.

#### DISCUSSION.

Mr. E. CAREY opened the discussion on the paper. He did not think it was possible to exaggerate the importance of the subject which Mr. Tate had brought before them that evening so elaborately and so well. He quite agreed with all Mr. Tate had said about the importance and also the difficulty of proper sampling. He thought that the sampling of goods in his own trade was bad enough, but Mr. Tate had described cases where goods in casks settled into layers, and the unfortunate sampler had to roll the casks about for a spell before he could get samples at all. It seemed to be a labour of hopeless magnitude to sample fairly in such cases. Merchants and traders paid very little attention to accurate sampling, and without accurate sampling reliable analysis was impossible. In a case he remembered some years ago, where a large quantity of copper pyrites required to be sampled, the percentage of copper varying from 2 to 3 per cent. up to 10 or 12 per cent., buyer and seller each agreed to pick out a lump in turn, and of course as each picked out lumps likely to suit his interests the results were widely different from the truth. Of course that would not be done in a place where they really understood copper ore sampling. The proper sampling of large cargoes was no doubt an expensive affair, but sampling could only be done in a thorough way when a large proportion of a cargo was drawn upon for sampling. He wished Mr. Tate had answered the question how many casks in a given lot of goods should be sampled—that was, what proportion. He regarded that as a very important matter. His idea was that a very much larger proportion should be sampled than was now the rule. Less than one in three he should consider a mistake; and he thought it would be well if analysts stated on their certificates what proportion of a given quantity of goods the samples were drawn from. It often happened that the seller asked the sampler to come and sample the goods at a time when he could only get at a small proportion, and if the analyst stated in his certificate how many samples he had received, or what proportion of the whole they had been drawn from, it would be a very desirable thing. A certificate at present might be quite right as far as it went, but it might not represent the whole; and if so stated no disappointment could be experienced, and sellers would take care that the samplers came when they could have proper access to all the goods requiring to be sampled. Seeing the enormous number of trades interested he was afraid that the proposed representative committee, if formed, would be rather unwieldy. It would be better if all the members or representatives of a few of the larger trades were to meet and formulate their views, and then submit them to some central committee. He repeated that it was impossible to exaggerate the importance of this question of fair sampling.

Dr. HURTER said that Mr. Tate had given them a very exhaustive account of the difficulties which beset sampling; and the result of hearing the paper had been to strengthen his view that it was impossible to give general directions as to how samples were to be taken; and the difficulty of arriving at a concise set of rules for drawing samples was very great indeed.

Mr. ALLEN said he was afraid that a general committee to discuss sampling in various trades would prove a council of words without knowledge. He would not care to be on a committee which would discuss alkali sampling, for instance; nor did he suppose that a palm oil merchant would be much at home with copper ores. He did not see how it was possible to lay down written rules and instructions



for sampling in all trades. A twenty-five years' experience of sampling a substance in which accuracy of sampling was of the very first importance convinced him that there were three requisites for the sampler—integrity, experience, and brains. Whatever class of goods had to be sampled, men of integrity must be had in the first instance. Much mischief was caused in times past through roguery in sampling, and therefore an honest, upright man was the first essential. The next requisite was experience. To send a young chemist without experience of the particular article was a mistake. A man with practical experience was to be preferred to one with scientific training without the experience. Then, finally, they must have men with right-down common sense. Given these requisites of integrity, experience, and brains they could take samples.

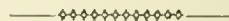
Mr. H. TAIT, jun., said it might be interesting to know that in the beetroot sugar trade they always sold on analysis. An association had been formed and a public sampler was appointed in London, in Liverpool, and in Greenock, by whom three samples of each lot of beetroot sugar were taken. One went to the sugar refiner, the other to the analytical chemist appointed by the association, and the third for reference. Thus all the samples passed through one man's hands, and he had a staff of samplers upon whom he could depend. Low sugars were not supposed to be sold by analysis. The association had only been formed two or three years, and it was found to work well. The samples they now got could be depended upon.

The CHAIRMAN said that where in the opinion of the analyst the sample was not a proper one, he ought to let that fact appear on the face of the certificate in some form or other. It would be easy to say that he had received such and such a sample, and that in his opinion it was not a good sample, or not in proper condition. For instance, if an article likely to give off moisture was delivered to him in brown paper instead of in a bottle, that fact ought to be stated, and its probable effect on the sample. He thought Mr. Allen had scarcely followed the argument as to written instructions. It was true that no written instructions could supply integrity, nor yet brains, but they would be useful, he thought, in imparting to others the experience of those who had gone before. He thought Mr. Carey had suggested the proper course to pursue—that was for a few skilled persons in the leading branches of manufacture to meet and draw up what they considered to be proper instructions both for sampling and testing, and submit those instructions to a central committee composed of one or other or all the three societies which had been named. He was sure they were all obliged and indebted to Mr. Tate for his paper.

#### REPLY.

Mr. TATE: As to the number of casks or parcels to be sampled in a cargo, cases had occurred to him where he had been able to draw samples from every single cask; and then there had been no disputes, the results of independent analyses being practically the same, and experience indicated pretty clearly that that was the best course of procedure. He saw no reason why the number of casks sampled should not be stated on the certificate, and he thought that if done, it would lead to some alteration in the mode in which parcels were sampled. He did not intend to suggest that the committee he had spoken of should lay down rules for guidance in every special case, but that such a committee should watch and press on improvements pertaining to sampling, and should have the power to nominate persons specially acquainted with certain trades and articles, to deal with any difficulties arising

in connection therewith, and draw up a set of rules for discussion and approval by the more general committee having charge of the whole question. Mr. Allen, he thought, had rather misunderstood him as to the employment of chemists as samplers. He agreed that it would be very wrong indeed to send a young chemist, an apprentice, perhaps, to sample a substance which he had never seen. Nothing could have been further from his mind; but chemists competent to analyse an article ought to be able to indicate how the samples should be drawn, and if necessary be able to draw them themselves. He was glad to hear what Mr. Henry Tate had said about the sampling of beet sugar; for that was the sort of thing he had had in his own mind. Here one trade dealing in one special substance had taken action to avoid disputes as to sampling; and by a very simple process appeared to have succeeded. The same or a similar system was what they wanted applying to a much greater extent. It was a practice which he almost invariably adopted himself, in analysing low sugars, to report the condition in which the samples reached him, and the probable loss of moisture, etc.; and that was what he thought analysts ought to do not only with sugar but with other substances.



#### DISCUSSION ON DR. HAMBURGER'S PAPER

May 7th, 1884.

MR. E. K. MUSPRATT IN THE CHAIR.

Mr. J. FENWICK ALLEN: Dr. Hamburger deserves our thanks for his interesting and valuable paper. The subject he has introduced continues to be one of practical importance to the manufacturer, and is one on which we fear there is still a widespread ignorance and prejudice. Damage to vegetation is often attributed to chemical vapours, which is solely done by natural causes. Since our last meeting, a gentleman has been writing to the *Manchester Guardian* complaining that Dunham Park is being destroyed by the chemical fumes from Widnes and Runcorn. It seemed to me so extraordinary that noxious gases should prove so destructive at a distance of 15 miles, that I went over to investigate the matter, and I found no proof whatever of the least damage being done by the chemical fumes from the Widnes district. Many trees are dying, but from manifest natural causes, the most dominant of which are:—

1. Poor, shallow, sandy soil.
2. Drainage.
3. Bleak aspect.
4. Exposure by removal of sheltering trees.
5. Hard winters.
6. Age, natural decay.

Some years ago a farmer complained of his hedges being severely injured by the smoke from St. Helens. There had been a storm from the west, and the hedges on the side exposed to the wind were quite brown; but it happened that there were four hayricks standing at several yards interval from each other. Portions of the hedge opposite these ricks were screened by them from the west wind, but were not screened from St. Helens, which lay considerably more to the north of west. Wherever the hedge was screened it was green, whilst the portions opposite the gaps between the ricks were brown, just as was the remainder of the exposed portion of the hedge. During the last few days I have collected these specimens (specimens exhibited) of blighted thorns and injured rose trees. Looking at the trees I should have said they had been injured by chemical vapours,

but they could have been affected by nothing of the sort; they were in full leaf, with abundant blossom in bud; but they were growing in an exposed position, and the cold cutting north-east winds had done it all. Had these been near Widnes or St. Helens no question would have arisen as to the cause of the damage—it would undoubtedly have been set down to the “chemics.” Last year I was at the railway station in Norton. Some of the timber there (as these photographs plainly show) has been much injured by the gases from Widnes or Runcorn. The station-master drew my attention to his garden. He said his celery had been considerably damaged: he attributed it to the smoke. The celery leaves over the whole crop were much injured: they were spotted and withering, but wherever there was a spot there was a grub, which in every case was busy at its work of destruction. The leaves of the parsnip were similarly affected, but not to the same extent. I have known heavy damages awarded for injury to celery by one of the fairest and most experienced of agricultural valuers. There is no doubt the manufacturer paid for the work of the caterpillar. Last year, in company with an experienced farmer and valuer, I several times inspected the country in every direction round Widnes. I found that where trees and hedges were utterly destroyed in almost every instance, the damage to crops was very slight, especially where the land was well farmed. It was the saying of an old gardener: “muck licks chemicks,” and there is much truth in it. The vigorous growth of well-matured crops withstand the influence of chemical vapours and suffer very little damage, whilst poor crops on badly tilled and impoverished lands are much more susceptible. Where crops are damaged by smoke, the injury is principally done on the portions of the field nearest to works, and not equally over the whole field. The acute damage which is occasionally complained of, is done, not by the average escape of gases, or by the average quantity of acid in the air, but by some accidental and temporary escape, caused by an insufficient supply of water, the breakdown of some apparatus, or the action of some workman. It is not quantity but concentration that does the great mischief. Large quantities of dilute vapour have little effect where small quantities of concentrated fumes are fatal. Dry gases, or gases evolved into a dry atmosphere, and passing over dry vegetation, do little or no damage; the same gases, in a moist atmosphere, or travelling over damp crops, leave behind them very visible traces. Dr. Hasenclever, in his admirable paper on this subject, mentions chlorine as the most prejudicial of all gases, then sulphuric acid, then hydrochloric acid, and lastly, sulphurous acid. I am inclined, from experiments and observations I have made, to agree in this opinion. I think the enormous quantities of sulphurous acid turned out from all manufacturing centres is so dilute, that but very little of it is condensed, and sulphurous acid is converted very slowly into sulphuric acid under ordinary atmospheric conditions. Dr. Hamburger very candidly told us he had introduced the subject, with the intention of showing that the chemical manufacturer was ordinarily accused of doing mischief which he considered should be attributed to others. Analyses made by himself and others showed that much less chlorine than sulphur is found on and in plants, and that, therefore, he considered the damage is mainly done, not by the chemical manufacturer, who only contributes the small proportion of chlorine and its compounds, but by coal consumers, glass makers, copper smelters, and others. He omitted to notice an important source of sulphur and a fruitful cause of damage at Widnes and St. Helens, viz., chemical waste. It emits sulphuretted hydrogen, this combines

with metallic substances deposited as dust from smoke:—first, sulphides are formed, and these are rapidly converted into sulphates; these sulphates corrode the leaves, and produce the effects shown in Mr. Hasenclever's book as the work of flue-dust. We are accustomed to notice on the roads, on the leaves of plants, and on newly-formed ice, the notorious blue deposit; on being tested this is found to be a sulphide, which in a few hours becomes converted into the sulphate, and this acts corrosively on the leaf.

Mr. A. E. FLETCHER: The subject which Dr. Hamburger has brought before us is one of great interest to all those engaged in manufacturing operations, particularly to those conducting chemical works. The difficulty of determining with certainty the cause of damage done to plants is often very great, and, as has been pointed out by Mr. Allen, the injury done by insects often resembles that done by acid vapours. There are insects which attack celery, parsnips, parsley, and doubtless many other plants, by eating out the soft part of the leaf between the upper and lower skins in such a manner as to be scarcely discernible, until their depredations are at an end. When this is completed, they make their escape, and burying themselves in the ground, leave the leaf to wither. Some insects attack the leaf from the outside, and whether by sucking out its juices, or by eating away the under side, kill the leaf without altering its form. Many of these cases, the general appearance of the plant whose leaves are withered, resembles one that has been injured by acid gases, the more so as the injury done by the insect is often that which weakens without destroying the leaf, so that the withering commences at the outer edge, as in the case of one attacked by acid. And not only does the action of insects often simulate that of acid gases, but the action of acids may resemble that of insects. I allude to cases where there are spots on the leaves: these are generally the work of insects, but may also be caused by gas. They may be so caused if a spot of soot or ash from a chimney falls upon the leaf. There fixed, it absorbs acid from the air, causing it to act locally, and if there is sulphide of hydrogen this, with oxide of iron in the ash, may form sulphide of iron, which, oxidising, will attack the leaf. I have proved that the formation of sulphide of iron does take place in this way, by scraping the surface of a frozen pond where shining spots appeared—shining spots like those that sometimes appear on leaves. The scrapings were collected and gave off sulphide of hydrogen on the addition of acid; they also were found to contain iron. Dr. Hamburger recommends the ultimate analysis of the injured leaves in order to determine the agent that has been at work. For my part, I doubt if such analysis is often of value. When I first became interested in these questions, I thought so too, and took much trouble in analysing the leaves of healthy and of injured plants, in order to discover a rule for guidance. I failed, however, to find any regularity on which to rely, and Dr. Hamburger's analyses point, I think, in the same direction. In analysis No. 17, for instance, we find chlorine 1.47%, the sample marked good; whereas, in No. 21, chlorine is 0.39%, and the sample is marked damaged; and throughout the list of analyses given no trace of a rule can be discovered. In many of the cases healthy plants are shewn to contain more sulphuric acid or chlorine than the damaged ones. If our lungs were to be poisoned and inflamed by noxious vapours, we should hardly expect to find, after death, traces of the gases which had caused the damage; just so in plants. The gases may be washed away before we can collect samples for analysis. Moreover, the proportion between the sulphates and chlorides is not constant in the same plant when healthy. In the



majority of cases analysis does not help us; often the damage is due to want of proper culture. It is always the weakly crop which succumbs first. There are many kinds of works which turn out smoke capable of damaging vegetation, among others I will refer to salt works. At Northwich and Winsford vegetation is decidedly damaged. When making some inquiries there I was much surprised to hear that the damage to the trees was done by "cats." This is the technical name for accumulations of salt in the flues, caused by leaks in the boiling-down pans. These "cats" give rise to the escape of hydrochloric acid gas, salt vapour, and chloride of iron, partly by reaction with the silicic acid of the hot bricks, partly by reaction with the hot sulphurous acid of the fuel gas. In the Cheshire Salt Works about one million tons of coal are burnt per annum in the form of the commonest slack containing much sulphur. There are numerous other works which give rise to escapes of sulphurous acid, such as smelting works, glass works, and alkali works. Then the question arises, which of the noxious vapours does most damage? Dr. R. Angus Smith made experiments by feeding plants with water containing sulphurous acid, or sulphuric acid, or hydrochloric acid. It was found that sulphurous acid did least, sulphuric acid did most damage. Now, when sulphurous acid comes to the plants, and is converted by oxydation into sulphuric acid, it is of course as injurious as if sulphuric acid itself. Hence sulphurous acid is an active agent of harm, and in very many districts the only source of harm, such as in the Black Country near Wolverhampton, in Sheffield, and in many other towns where chemical works are not frequent. In Liverpool also the sulphurous acid of coal smoke is generally the damaging agent. I have often been asked how it is that the vegetation of London escapes comparatively well. Two reasons can be given. First of all the climate is much better, there is much more sunlight, high gales are not so prevalent as in Lancashire, and the air is warmer. Secondly, London is not nearly so smoky a town as St. Helens for instance; in proportion to the area, there is ten times as much coal burnt in St. Helens as in London. And in St. Helens coal is burnt all the year round, whereas in London it is burnt chiefly in the winter, when the leaves are absent. Plants are damaged chiefly by damaging their leaves; also the bark, not so much by the materials in the soil, indeed plants can live in very differently constituted soils. A very curious question had lately arisen as to whether plants had been damaged by absorption through the roots, or through the leaves. In a certain lead works there had been accumulated a heap of lead fume from flue cleaning. By a flood this heap was carried on to some adjacent field, and the grass poisoned. An action was commenced, and damages had to be paid. But later on the cattle eating the grass again were poisoned, and it was alleged that this was caused by lead fume coming from the chimney. The question arose whether it was the remnant of lead carried by the flood on the land, which caused the damage, and for which damages had already been obtained, or whether it was the fresh lead fume from the chimney which had now spoiled the grass, in which case new damages would have to be paid. Very interesting is the way in which manufacturers have striven to diminish the escapes. The escape of hydrochloric acid has been brought down from 5% to 1%, and the amount of acid per cubic foot has been reduced to a very small amount. In 1870, 61 furnaces had to be stopped in the western district under the alkali act on account of leaks, whereas the number now stopped is very small, owing to improved construction. The setting of decomposing pots has been improved also. It used formerly to be possible for the acid gas to leak into the flues

and chimney, now they are so set that this can scarcely happen. I was very much interested in Dr. Hamburger's paper, and thank him for having brought it before us.

Dr. HAMBURGER: I am sure we are all greatly obliged to Mr. Allen and Mr. Fletcher for having given us the benefit of their extended experience. I quite agree with these gentlemen that meteorological influences and atmospherical changes are the most important causes of damage to vegetation, although I did not treat the subject from this point of view, having termed my paper "On injury done by gases arising from certain manufacturing processes." I also know, that in exposed places, in Knowsley Park for instance, there were trees at the border much injured, whilst not ten yards away but nearer to the middle there were fine, healthy trees. In Garswood Park the same thing is visible. Now Garswood is exposed to the smoke of both St. Helens and Wigan, and the highest trees are injured, while trees in the centre not so exposed are not affected at all. Was that damage been done by smoke? Decidedly not; for smoke would not only have injured the tall trees but all the trees in this neighbourhood. There you can see striking instances of damage done by piercing winds. The trees are partly destroyed whilst shrubs in the immediate neighbourhood were almost free from acid. Mr. Fletcher has alluded to causes why the South of England is better off with respect to vegetation than the North. But it must not be forgotten that the soil in the south is better than the soil of Lancashire. Both Mr. Allen and Mr. Fletcher have alluded to insects and the similarity of appearances of damage by insects or by smoke. I have often noticed this on the rhododendron. Mr. Fletcher has also referred to the impossibility of judging from the analysis of plants as to the cause of damage. I quite agree with him; my analyses show no such rule. In conclusion I wish to impress once more the importance, never to conclude that injury to vegetation was due to smoke or noxious gases, until we have satisfied ourselves that it was not due to any of those natural causes. The damage which can be clearly traced to chemical works in the present way of working is very small indeed compared with that done by causes over which we have no control.

Mr. ALLEN: Would not simply washing the leaves and analysing the washings do?

Dr. HAMBURGER: I think decidedly not, because we do not know then how far lixiviation beside surface washing has gone on. If we wash the leaves and afterwards ignite the residue and add the results, obtained in both ways, the process would be satisfactory. However, ignition with an alkali to prevent the escape of sulphuric acid and chlorine is a great deal simpler.

The CHAIRMAN: The remarks made by Mr. Allen have tended to show that there are natural causes of damage to vegetation which at first sight may be mistaken for damage caused by chemicals. The photographs which Mr. Hasenclever published shewed very distinctly how trees damaged by drainage had very much the appearance as if damaged by acid gases. Want of water was very often the cause of decay. An impervious pavement, if carried too near to the roots of a tree will often kill it. In Liverpool where streets were made near trees, it happened frequently that an apparently healthy tree died almost suddenly. If the streets or pavement round or near a tree are asphalted or trodden down the tree is deprived of water and decays. On the Prince's boulevard in Liverpool attempts to grow trees failed in the first instance from this cause assisted also by the strong wind to which the trees there are exposed. Mr. Fletcher has referred to salt cake furnaces and assigned as a reason why he had not to stop so many

now as formerly, that these furnaces were now constructed very much better. Now he was not aware of any radical change in the construction of the furnaces, but there was no doubt that now in times of quiet trade the artisans attended much better to the little details of construction than they could do in times of brisk trade such as 1870. As regards the pans; the people who cast the pans and professed to cast good pans, had most certainly improved the quality very considerably so that cracks and leakages are certainly of less frequent occurrence. As long as a good quality of iron is used, pots last a reasonable time; but when iron is dear and inferior iron is used then the pots break.

## Manchester Section.

Chairman: Ivan Levinstein.

Vice-Chairman: George E. Davis, F.I.C., F.C.S.

### Committee:

F. B. Benger.  
R. F. Carpenter.  
C. Estcourt.  
H. Grimshaw.  
Peter Hart.  
J. von Hohenhausen.

T. Jackson.  
S. Mellor.  
C. Schofield.  
R. Angus Smith.  
W. Vickers.  
D. Watson.

### Hon. Sec.:

J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

## MANCHESTER TECHNICAL SCHOOL,

June 10th, 1884.

## ADJOURNED DISCUSSION ON STANDARD METHODS OF SAMPLING, ANALYSIS, AND STATING THE RESULTS.

The SECRETARY read the Report of the Committee.

### MAY REPORT.\*

IT has already been decided by the last meeting "That in the opinion of this meeting it would be practicable and very desirable to attain a national agreement on standard methods of sampling ores, raw products and chemicals; but that an international method would be at present impracticable."

The Committee were also requested by the last meeting to carefully consider the discussion which took place, to examine the propositions put forward by the several speakers, and to prepare a series of resolutions for the May meeting, incorporating the gist of the discussion.

Your Committee has met to consider the matter, and begs to report as follows:—

Question 1 has been practically disposed of so far as national methods of sampling are concerned, by the resolution of the last meeting.

2. In reply to the question "Does a national agreement meet all purposes, at least so far as this country is concerned?" Your Committee is of unanimous opinion "That a national agreement would serve all purposes," this conclusion being arrived at from carefully considering the report of the conference of April 1st.

3. With regard to the third question "What are your opinions concerning uniform methods of sampling, and how could an agreement be arrived at?" Your Committee has been unable to arrive at a unanimous conclusion, and therefore recommends further discussion upon this subject.

4. To the question "What are your opinions concerning uniform methods of analysis?" Your Committee are not unanimous in their conclusions, but considers that chemists may use the best known methods, and should be asked to state the method used upon the report or certificate of the analysis or assay.

5. As to uniform methods of stating results, your Committee is of opinion that such would be possible, and invite further discussion on the subject.

6. As to the appointment of Committee and their powers, your Committee is of opinion that such a question may safely be left to the Council and a general meeting of the Society.

7. In reply to the question as to whether the adoption of standard methods should be made compulsory, your Committee is unanimously of opinion "That any agreement which may be arrived at shall only be considered as a recommendation to chemists, buyers, sellers and consumers, and not be absolutely binding on the parties concerned; and it feels sure that the publication of the best methods in the Journal of the Society, and examined from time to time by a representative committee, would in course of time have considerable influence in securing their adoption."

Mr. MELLOR: It appears to me that the method of sampling must greatly depend upon the articles to be sampled. Clearly if we are sampling minerals, or metals, or alloys of metals, or metals containing gold or silver, or articles in solution, the method of sampling in each case must vary according to the article to be sampled. Now, I submit that small sub-committees should be appointed, consisting of gentlemen accustomed to deal with the sampling of given definite classes of substances, whose duty it should be to draw up suggestions of methods of sampling of the list or class of articles with which they are best acquainted. The reports and methods suggested by these sub-committees could be discussed *seriatim* and be altered, improved, and ultimately adopted by a full meeting of our section. Any method of sampling drawn up with such care, and approved by our Society, would soon have the binding influence of the law of usage, and would be adopted generally. To avoid disputes and to save the reputation of analytical chemists in all contracts between buyers and sellers, the method of sampling should be agreed upon and stated, and in the case of minerals and valuable ores or "precipitates," the mode of ascertaining moisture should be given, and always it should be determined if the metal contents of ores or precipitates is to be ascertained by fireassay, or by complete analysis. I propose that the subject be referred back to the Committee for re-consideration, and that their report be brought up at the next meeting.

Mr. GEORGE E. DAVIS said: Mr. Mellor's remarks have, to my mind, proved two points—firstly, that a heterogeneous committee would be incapable of dealing satisfactorily with the questions in detail. When the details were worked out, the general principles might be settled by a larger committee. What would be the use of an anthracene maker endeavouring to draw up a method for the sampling of minerals, or a refiner of the precious metals being consulted as to the analysis of benzol or dyewood? To my mind the process is now definite; the Committee of this Section, having the results of the Conference before it, has been of the opinion that the appointment of a general committee to consider the subject would be a

\* This report was referred again to the Committee.



step in the right direction; such a committee could appoint sub-committees of the special trades upon the questions of sampling, stating the results of the analyses of those special articles which came under their notice, and the analyst would still be left to choose his own process, and be responsible for the accuracy of his report. Secondly, I think Mr. Mellor has also proved that the sampling process is a most important one, that it should not be left to uneducated persons, and that more prominence should be given to it in the future. On behalf of the Committee, I have no hesitation in saying that it will only be too happy to make the report more definite in the direction spoken of by Mr. Mellor if the meeting desires it.

Mr. TYLER said: I should like to supplement Mr. Mellor and Mr. Davis's remarks by relating how matters stand in the Metropolis. I need not go into the history of the matter; I wish only to say that with regard to this particular Section I am in absolute accord, and I believe I may say that every business man and every manufacturer with whom I have conversed agrees absolutely with the views which were expressed so practically at the last meeting. I don't want to apologise for what might appear to be an indefinite conclusion in the London and Newcastle Sections. If you recollect, these resolutions seemed to commit us to nothing. The discussion covered a large area, but there was a certain amount of indefiniteness in it. It was said by visitors on that occasion, who were eminent as analysts, that they felt the enormous difficulties of the subject.

We are getting with the practical men of Manchester to something like a practical resolution. Now the aspersion upon the analytical chemists has ever been too freely cast, and the time has come when the methods of analysis shall be such that the chemist may attach his name to the results and appear, if necessary, with his reputation in his hand in the witness-box. If there is a difference between A and B, it must be either inefficiency of method or a vital difference of methods. But there is the difference of samples.

If two chemists agree as to methods, I really see no reason why there should be any disputing their results, assuming—and I agree with what Mr. Mellor and Mr. Davis have said—that we have committees. The London Chamber of Commerce has two chemists on the committee, and this body issued a circular inviting the co-operation of the several trades taking an interest in the subject, and the Chamber did credit to itself as a mercantile body in tackling the subject. The Chamber in effect said: "We have our Manure Manufacturers' Association, and they have their own referees. If there is any doubt, we appoint our two chemists, and if they don't agree we appoint a referee." The Manure Manufacturers' Association appointed a committee to consider the subject, and a resolution was proposed to sample a heap of superphosphates, and they took samples from the same heap at the same works and sent the sample to five analysts chosen from the Directory, without knowing anything about them, and the result may be learned from the following report:—

"At a meeting of the representatives of the Chemical Trades, which included, by invitation, non-members as well as members of the Chemical Trades Committee, held on March 14, the following resolution was passed: 'That the professional chemists be invited to meet and consider what means, if any, they can suggest by which the most accurate mean result may be arrived at, by any two chemists who may be named, in any phosphate or superphosphate contract.'

"At the first meeting, on April 2nd, Mr. Ogston was asked to take the chair, and after a full discussion of all the matters relating to analysis and phosphates that had been mentioned at the various

meetings of the Section, and having particular regard to the statements made by many of the representatives of the chemical trades, at the meeting held on March 14, it was proposed, and seconded, and resolved that before discussing any differences of methods employed by the several members of the sub-committee, it would be desirable to ascertain what variation would be found in the analysis of a properly prepared sample when made by the several analysts. Such a sample of Charleston phosphate was prepared by two members of the Committee, and a portion sent to each one for analysis by their ordinary methods. The results were compared at their next meeting, and the following figures represent the five determinations of phosphoric acid:—

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Phosphoric Acid - - - -	26.55	26.20	26.48	26.60	26.00
Equal to Phosphate of Lime	57.96	57.19	57.82	58.06	56.75

"It will be seen here that the differences are not large, and probably do not vary to a greater extent than may be expected in substances of this character, and considering that in all cases the mean of two analyses is taken, the variation cannot be said to be of any serious importance. Upon discussion it was found that in four cases out of the five the 'Magnesian' method alone had been employed in the analyses, and that in the fifth the mean of one test by that method and another by the molybdenum process had been taken, the difference between the two being one-half per cent. in the phosphoric acid.

"It would appear from these results of the analysis of a phosphate—by no means of the simplest composition—that fairly uniform percentages of phosphoric acid are indicated, and that there is uniformity of method in the practice adopted by the reporters, and as they believe by all the principal analysts engaged in the examination of phosphates in this country. The sample that was reported upon was, of course, prepared very carefully, but not more carefully than samples should always be dealt with before being forwarded for analysis, and in reference to this part of the question it was agreed:—

"1st. That the manufacturers should be asked to send the ground samples in a very finely divided state, with a separate sample coarsely crushed, in which the determination of the water shall be made.

"2nd. That the phosphoric acid shall be determined in the ground sample dried at 212° Fah. The moisture to be separately estimated in this, if it be desired, as well as in the rough sample.

"3rd. That in the event of there being a difference of more than 1½ in the results of the two chemists in the phosphate of lime, these results shall be communicated to both of them, and their opinion requested as to the cause of difference.

"It will be seen that the extreme difference in the five analyses above referred to is 1½ per cent. in the phosphate of lime, and it is thought that it should not exceed 1½ per cent. in samples finely ground and prepared as we recommend.

(Signed)

"G. H. OGSTON,  
"JOHN HUGHES,  
"H. R. SMITH,  
"BERNARD DYER,  
"F. E. J. CRIDLAND."

Mr. JAMES RICHARDS\*: No one who has given careful attention to the subjects we have to consider to-night, can doubt the importance attaching to a full discussion of it in a society like our own, nor the desirability of a national and international agreement

\* Mr. James Richards had prepared the following statement but time not allowing, it was resolved to take it as read.

on the first three questions if practicable. The different results obtained by chemists of high standing in their profession, are at times so great, that one is not astonished when one is sometimes told by impetuous non-professionals that analysis is all humbug, and it is as necessary to the reputation of the scientific chemist as it is to the pocket of the manufacturing chemist that these differences should be reduced to a minimum. I remember a cargo of guano coming into Liverpool some 20 years ago, which was sold on a basis of about 11% ammonia, and I analysed a sample sent by a firm of brokers there, which shewed rather more than that, nearly 12%. We bought, I think, 100 or 150 tons. The first lot that arrived at our warehouse contained 8½% of ammonia. We complained, and a sample of the lot was sent to Dr. Anderson, of Glasgow, who reported it rather higher than I, about 8¾%; we got the allowance we claimed. This was bad enough, but a later lot only tested 5½%: the brokers maintained my analysis was incorrect, but as we refused to unload it, they at last sent a representative to sample along with me, and the sample was divided into three portions. One I analysed, finding 5¼%, another was sent to the doctor, the third sealed by the brokers' representative, and kept by myself. They allowed our claim, paid £20 demurrage, but would not give me the doctor's figures, merely saying they were higher than mine, but their allowing the claim shewed the substantial correctness of my figures. On investigating the matter, the only conclusion we could come to was that the carbonate of ammonia found in the top part of the cargo was due to the decomposition of the nitrogenous matter of the guano in the lower part, owing to the latter having become damp from contact with the bilge water of the vessel. The sample in this case had evidently only been taken from the top of the cargo by someone who did not understand his business. But apart from such a gross case as this, the results of analyses of shipments of superphosphate shew variations so great that one can scarcely believe that any differences of methods employed by efficient chemists would account for them. On a shipment made by my firm, Keames, Richards and Co., last half-year, we had the following figures:

Phosphoric Acid, determination	Own	London Chemist	Liverpl Do.	1st American.	2nd American.
Soluble in water..	12.25	12.32	13.78	11.78	11.50
Do. reverted ..	2.29	1.63	.45	1.09	2.04
Total available ..	11.54	13.95	14.23	12.87	13.54

We had to receive payment on the lowest of the four, making a difference on the shipment of £450. Between the highest and the lowest there is a difference of 1.67%, or calculating the highest as 100, a difference of nearly 11½% of available phosphoric acid. The two English chemists did not determine the insoluble phosphoric acid, but the following figures are the results of our own, and the two American chemists respectively, of the total phosphoric acid:

Own.	1st American.	2nd American.
15.98	16.63	16.33.

Or taking the highest American as 100, we get own 94.88, 1st American 100, 2nd American 98.2, or an extreme difference of 5.12%, and both the latter higher than our own. The only remark I have to make on this is that nothing is paid for insoluble phosphate. I will give you one other example which occurred last month. The London chemist already mentioned found in a sample soluble in water 11.76, reverted 1.65, total 13.41. Two American chemists (in neither case the same as before mentioned) gave the following figures: No. 1, total 12.98; No. 2, soluble in water, 11.38; reverted, .31; total, 11.69. The latter chemist, our agents tell us, has a great reputation abroad, and the result was arrived at by him after three careful analyses. The singular part of the business is that the doctor's method is exactly

the same as we employ ourselves, with the exception that he uses 2 grammes of superphosphate where we employ 5, and digests at 40° C for 30 minutes, while we take 45 minutes (the larger quantity taken in our case accounts for the longer time required); and while we grind the sample fine, "he before testing does not pulverise the sample in a mortar, which some others do, which he claims is not proper, as the goods are not pulverised by the buyer." That is to say, the learned doctor does not apply himself to the question put before him, the amount of available phosphoric acid in the sample, but to a totally different problem, how much available phosphate he can get out of a given quantity in a limited time, with a limited amount of solvent under certain undefined but certainly not the best physical conditions for its extraction. One can understand a firm of high standing and large experience, but with some natural shrewdness, declining to describe their method to Mr. Allen, or any other chemist, when the head of a state agricultural experiment station can give such a peculiar reason for such a peculiar method, and get such peculiar results, and you can understand the remarks of our agents who are not chemists, "the trouble is that no two chemists use the same methods, and consequently all differ;" and again, "unless there is some uniform method adopted here (that is America) and abroad, every lot is subjected to this difference, and we believe the only safe way to sell goods is on a soluble basis, making prices accordingly either by English (naming chemist) or American analysis. To sell on English chemists' analysis, if available, is no longer possible, as buyers are now aware of the difference made by the chemists, and will not risk the almost certain loss to themselves." I think, gentlemen, these examples tend to shew the absolute necessity there is for this society to use all possible endeavours to attain an international agreement on standard methods of sampling and standard methods of analysis or assay whenever a large industry is concerned. The question of specification of method of analysis in the contract, as suggested by the Chairman of the London Section, is one of great importance, and one that ought to be taken up very seriously by the artificial manure manufacturers; and I do not think that the opinion of chemists of high standing is the all-important factor in the discussion of the business aspects of the case. What we want is a recognised commercial value for certain definite chemical results attained, such results to be ascertained by a clearly defined method, the most exact and widely recognised that the present state of science can furnish us with. And by most exact I mean not that which is the nearest to the absolutely accurate, but that which shews the truest relative correctness, in which the comparison of results can be most surely relied on. I think with Dr. Hurter that, in order to attain this, it is necessary to decide whether it is possible to fix upon unalterable units, and I agree with him that it will be possible to devise units that will need no revision. With regard to the method of returning the results of analysis or assay, I cannot see the force of some of the objections put forward, as if invariably returned on the centesimal system, each individual would soon learn to convert the results into the form he is most familiar with. The tendency is now in this direction, and it does not at all depend on the adoption of the metrical system in any other department of business. I consider that a committee of this Society, consisting of representative, professional and manufacturing chemists, and brokers and consumers from, and appointed by, each Section, should be formed to ascertain, as far as possible, the views of those interested in the various chemical and allied trades of the district, whether members of this Society or not; and



that after conferring together these committees should present their report to the Newcastle meeting, where a smaller committee might be appointed to confer with the Chemical Society, and the Society of Public Analysts, with a view to the furtherance of the solution of the questions we have this evening for discussion.

## ON THE MANUFACTURE OF WHITE LEAD, AND GARDNER'S ELECTRIC WHITE LEAD.

BY J. CARTER BELL, A.R.S.M., F.I.C., &C., &C.

The manufacture of white lead is a most ancient proceeding, and has been pursued with but little variation in the mode of manufacture for some hundreds of years. The Dutch seem to have been the originators of this method of making white lead, which is now so largely conducted in this and other countries.

In this process metallic lead is piled in stacks, and submitted to the action of acetic acid, watery vapour, air, and carbonic acid for some time, by which means the metallic lead becomes gradually converted into white lead.

This method is called the "Stack" or Dutch process.

The construction of a stack is a very simple and rude operation. Layers of dung or tan, or a mixture of the two, so arranged as to imbed a large number of earthenware pots, each containing some acetic acid. These pots are about 4 or 5 in. in diameter, and about 7 or 8 in. high; a coil of lead is placed in each pot, and buckles or gratings of lead supported on oaken bearers are laid across and on top of the pots; boards are laid to cover the whole, and form a floor.

The stack is composed of a number of such layers of pots, bearers and buckles or gratings, raised one upon another.

A stack chamber is a brick inclosure 10 or 12 ft. square, and 20 or 25 ft. high; such a chamber will contain about 70 tons of lead when stacked and piled. In a white lead factory several of these chambers are built side by side, and when they are in full operation a set of chambers will contain as much as 700 or 1000 tons of lead.

Only the purest kind of lead will be suitable for conversion in this stack process of making white lead, the common varieties being inadmissible. Messrs. Pontifex and Wood have kindly given me the following analyses of lead used for white lead making.

	Copper.	Antimony.	Iron.	Spelter.	Silver.	Lead.
A	0.00700	0.00410	0.00200	0.00080	0.00100	99.98430
B	0.07580	0.00320	0.00220	0.00320	0.00200	99.91360
C	0.00340	0.00460	0.00120	0.00070	0.00350	99.98660
D	0.05260	0.00740	0.00150	0.00180	0.00400	99.93270
E	0.00640	0.00210	0.00160	0.00100	0.00075	99.98515
F	0.02360	0.00580	0.00210	0.00180	0.00100	99.96570

The presence of silver, copper and iron in the lead would damage the colour of the white lead resulting, and other admixtures retard or prevent the progress of conversion.

In olden times horse dung was the only imbedding material used in the stack arrangement. This material when heated evolves gases which seriously interfere with the colour of the resulting corrosion. Dung has been almost superseded in this country by tanner's refuse; in Belgium dung is yet employed, and in some places a mixture of dung and tan.

Where dung is used, the process of corrosion of the lead goes on more quickly than when tan alone is employed, but the use of tan offers great advantages, especially this one: that it does not give off gases that damage the white lead.

The operation of charging and discharging these chambers is principally the work of women, and is most laborious and fatiguing.

In emptying the chambers and stripping the stacks the women are fully exposed to the heated gases which are yielded by the decomposing tan, and the heated and corroded lead. These gases, in themselves most injurious to health, are not to be compared in this respect to the dust which pervades the air and fills the chamber in which these women work.

When a stack is charged, the chamber containing it is enclosed. The tan or dung within soon commences heating, and the heat soon causes the acetic acid in the pots, and the water in the tan or dung to rise in vapour and penetrate the stack. Air is admitted to the stack through openings left for that purpose, and carbonic acid is evolved from the heated decomposing tan or dung, and this gas also penetrates the stack, and the process of converting blue lead into the white lead gradually proceeds, and the blue metal becomes corroded and incrustated with a white crust or covering.

As to the exact chemical changes and combinations proceeding in the working of a stack differences of opinion exist, but we may fairly conclude that the process resolves itself into this—first, the formation of sub-acetate of lead, which, decomposed by the agency of the carbonic acid gas, becomes reduced to the condition of normal acetate by loss of a portion of its basic oxide of lead. The reduced sub-acetate then again takes up an additional molecule of oxide of lead and is re-converted into its original subsalt state to be again attacked and reduced by the carbonic acid gas, and so on continually during the working of the stack.

It will be evident that the "nascent" state of the various substances engaged during the chemical changes which are proceeding in the stack is an important factor in this process, and must be taken into account in considering the philosophy of the operation.

It will also be evident that the mode of proceeding in white lead making by the stack process is most crude and clumsy, and a most uncertain method, one governed by rule of thumb, and by no element of certainty or science. White lead makers, as a rule, know nothing of the chemistry of their subject. This absence of chemical knowledge of the subject, by those who are engaged in this manufacture, may explain the curious circumstance that for hundreds of years this industry has been pursued in the same old-fashioned and uncertain way, and the stack, or Dutch process, still holds its ground and displays little or no advance in knowledge or improvements in its method of proceeding, even in the present age of precision in almost every branch of manufacture. The uncertainty of the stack process is shewn most clearly in this: That stacks may work and some do not work. In the latter case all the time and labour spent in forming the stacks, and all the acid they contained, is lost.

No amount of foresight will avail to determine beforehand which stack shall accomplish the conversion of its contained metallic lead, and which will not.

The stacks are generally allowed to remain in operation, after they are charged, three or four months; in this time it is presumed all profitable action in the stack has ceased. The temperature of the stack which had risen gradually from the normal temperature to 100° or 150° Fahr. will have gradually fallen, and this falling temperature is the indication that the corrosion of the lead in the stack has terminated.

After the three months' action of the stacks, they are stripped and pulled to pieces. Some will be found

to be done better than others, and one part of the same stack will be done more perfectly than another. The coating on the lead will also differ; some will be smooth, regular, and equal in formation, some will be rough and blistered, and very ununiform. The rough blistered casting is rejected as unfit for white lead making, the workmen call it "dross." The smooth laminated coating is the one preserved for the after manufacture. It is a curious fact connected with the consideration of the total want of educated guidance in these matters that prevails, that in all factories of this description some chambers are noted as always working well, and others are equally well known to always do their work the reverse of well. No one knows why! No one stays to seek the reason. The factory way goes on filling and emptying these white lead chambers whether the stacks be working well or no.

The incrustation that is most esteemed by the manufacturers of white lead in this old-fashioned way is a hard, china-like material, formed of thin deposits, layer upon layer, in a slow, continuous, regular way. It is at once conceivable that in the rough-and-ready manner of stack manufacture most irregular action must proceed.

It would be almost impossible for the contents of the pile or stack to be submitted to the same action of the gases throughout. Some part of the stack and its contents will be under more favourable conditions than another, hence the reason why, in practice, it is invariably found that some stacks, and some parts of a stack, work better than another. Under the microscope this good crust of white lead, the proper incrustation from which to prepare white lead, will be readily seen to consist of very thin coatings or layers of white lead, which have been slowly formed on the metallic lead and piled one upon another to the thickness of an eighth or a quarter of an inch. This formation constitutes the hard, china-like substance, which alone possesses the chemical constitution and the properties to form good white lead paint.

White lead makers, recognising this peculiar incrustation as the only one capable of fulfilling their desired purpose of making good white lead paint, do not even recognize any other as of any service for that purpose, be it good or bad. Such a material, if obtained, however good, would be outside their experience and beyond their philosophy. After the stacks have been stripped, the gratings or buckles with their adhering coating of white lead are moistened with water and are passed through crushing rollers to separate the unconverted lead, then the crust which has been detached is ground under heavy edge runners with water.

This detached crust of white lead will vary much in colour, it will be white in some parts, yellowish or greyish in others. These discolourations arise from various causes, but they are principally caused by the contact of the moist wood and tan. The white lead is now a rough crushed material, very hard, and requiring to be ground to the finest powder. It contains, also, small fragments of blue lead, which have passed the crushing rolls, and a quantity of acetate of lead. The presence of acetate of lead is always found in larger or smaller quantities, which vary with every operation, and which invariably accompany white lead produced in the stack.

To remove discolourations—to separate the fragments of metal and to dissolve out the acetate salt—much water and washing is employed. The material is ground with water under the heavy edge runner stones, it then proceeds to a series of horizontal mills, each succeeding mill set closer than its fellow, and is further and further ground to fineness with water. From these mills it runs as a milky liquid to a series of setting tanks where it is allowed to subside, and

the clear fluid run off to waste, or into tanks to be used over again. This waste water will now contain the colouring matter removed from the incrustation, and the principal portion of the acetate of lead which the incrustation previously contained, and any other soluble matters removed from the washed and ground material. The small fragments of lead which passed the crushing roll and edge runner mills will have been previously removed by subsidence in water.

The white lead deposited in the tanks is in some factories ladled out into skips and agitated by a "Dolly," which further enables the heavy powder to get free from the water in which it is entangled. The moist powder is next placed on trays or dishes, and is conveyed to the stove or drying chamber. Women always perform this work.

The drying or stoving room is a large enclosed space heated by a "Cockle" arrangement; rough scaffoldings are erected within this chamber on which women mount to stow the trays on shelves fitted for the purpose. The trays and their contents remain in the heated atmosphere of this chamber for two or three weeks, by which time they become dry and ready for removal, to be packed in lumps for certain markets, or ground to dry powder and packed in barrels for others.

Women are employed to fill and also to empty the drying or stoving chamber, and during this work they are fully exposed to its contaminating atmosphere. Hot and dry, and charged with fine dusty particles of white lead, it becomes a dangerous trap, and contaminates the blood of those engaged with its deadly poison. It is in this part of the manufacture that the principal damage to health occurs. This is the most laborious work; heat makes it very fatiguing, the atmosphere within this chamber being always much above the exterior air. Recent Government regulations have sought to curtail these and other evils in this manufacture. Women engaged in these stoves are ordered to wear overclothing, headress and respirators; the general experience of their practice, notwithstanding Government regulations, is this: that they cannot work in them with ease and convenience, and more often wear the respirator around their necks than in front of nose and mouth. The excessive mortality in women who work in these stacks and stoving houses scarcely requires reassertion. Few, even of those who employ them, know the extent of the deadly operation. Recently, medical men have made public that cases are within their knowledge of children born already contaminated with lead poison. Woman labour should surely be restricted by Government enactments in all such deadly occupations.

We may sum up the whole matter as regards white lead making by the stack or Dutch method in a few brief words: It is a most tedious and uncertain operation: it is a most dangerous occupation for all concerned; it is founded upon no true principles of any kind; and of science its whole course is ignorant. White lead making is ruled by a "happy-go-lucky" philosophy. The representatives of this manufacture are completely ignorant of the scientific details relating to it, and hence we may not be surprised to find amongst them an enormous amount of ignorance and prejudice.

Good white lead will not differ materially in its composition by whatever process it may be made, but it may differ seriously in its physical character, and in its fitness to produce a substance adapted to the uses to which white lead paint is applied. Good white lead is a compound which contains hydrate and carbonate of the metal, in the proportions either of one molecule of hydrate of lead, combined with two of carbonate, or is made up of one molecule of hydrate, with three of carbonate of lead.



If we consider the first compound roughly  
 $\text{PbH}_2\text{O}_3, 2\text{PbCO}_3$   
 white lead will be made up of one part of hydrate over two parts of carbonate of lead.

The second compound roughly estimated  
 $\text{PbH}_2\text{O}_3, 3\text{PbCO}_3$   
 will be one part of hydrate, combined with three parts of carbonate of lead. The latter will be in the proportion of 75 per cent. of carbonate and 25 per cent. of hydrate of lead, and this represents the composition which has been assigned to good white lead by those most acquainted with the subject. The amount of hydrate contained in white lead should never exceed the proportion above named of 25 per cent., nor should its amount be much below the 25 per cent.

The hydrate contained in the substance serves to unite with the oil in the paint, it forms therewith a drying white and elastic varnish which embraces and holds the particles of white carbonate and prevents their subsidence and separation in the paint. There is a chemical action of a much more intimate character between the components of good white lead when mixed with oil which neither of the constituents of this compound can alone produce.

For instance, hydrate of lead and linseed oil produce a varnish-like substance semi-transparent and of no covering capability.

Carbonate of lead and linseed oil produce a compound which is opaque, but has no body or covering power and in which the white solid carbonate is held in feeble mechanical suspension. Neither of them constitute a paint, but when together as white lead they are mixed with oil combination takes place, and serviceable paint of good body and covering power and enduring quality is produced. Good white lead is a dense, perfectly amorphous powder of perfect whiteness possessed of great body and covering power when combined with oil. When mixed with linseed oil and used as paint it rapidly dries in the air and assumes a varnish-like, glossy, hard surface, and is capable when once dry of resisting the action of air and water for any length of time. It does not weep when laid on a surface with a brush, that is the oil does not separate from the solid material of the paint.

Attempts have been made to produce white lead quickly and cheaply by precipitating processes, but in all such methods the resulting compound is deficient in certain special qualities absolutely necessary to white lead proper and to its uses. The precipitated white lead is always of a crystalline structure and crystalline lead can never furnish a good body paint—no amount of pulverizing and grinding of this crystalline material will correct this defect in its nature, and deprive it of its crystalline form.

"Once a crystal always a crystal" has an especial application to this point of our philosophy. Pulverising a crystal will not alter its structure, but simply reduces the size of the crystals. Crystals of white lead are unable to effect the necessary combination with the oil and form the true varnish which white amorphous lead so readily produces. Paint made with the precipitated white lead lacks body and covering power, and this because of the absence of this chemical union with the oil.

The manufacture of white lead by process of precipitation, even were the resulting preparation suitable, does not correct the evils of the present method by Dutch or stack process of making white lead.

A solution of lead may be precipitated in a few minutes, but it cannot be made so quickly. The white lead after its precipitation has to be filtered, or separated, washed and dried, and ground to powder, which processes cannot occupy less time than a few weeks for completion.

Precipitated white lead has been made in France and Germany for some years, and it is now manufactured in those countries. It is now made in England by one patent process, but the product lacks certain qualities, and is consequently still open to the objections already noted.

Substitutes for white lead of a non-poisonous nature, or of such a nature as not to produce such deadly effects in their preparation or use as white lead does, have been proposed; their introduction has not, however, been a great success. I may mention some of these—Albissima silicate paint, patent white sulphide of zinc paint, and zinc white. They are known to us and are—

Ground silicates and oil and driers.  
 Oxychloride of lead.  
 Sulphide of zinc.  
 Oxide of zinc.

A mixture of sulphate, sulphide and oxide of zinc is a patent white made by subliming galena in an oxidizing furnace or hearth. This compound lacks body. All of these so called substitutes are very inferior to white lead, not only as to quality but as to cost. They cannot compete with white lead. A committee of enquiry on these substitutes for white lead, reporting the result of their enquiry and examination, stated that they found that these were mostly prepared with varnishes before they were sold for use, and that in most instances were mixed with a large quantity of driers, and that the drier invariably was a compound of lead. The principal consumption of white lead is for paint; to produce this paint it is ground with oil in varying proportions, about 8 or 15 per cent. This produces the ordinary white lead in oil, and is worth from £19 to £20 a ton, but often more than this amount.

Dry powdered white lead is chiefly made for and used by grinders and mixers, who combine with it a variety of other cheaper materials—chalk, clay, sulphate of lime, and sulphate of baryta, but principal use is made of chalk and barytes. These are mixed with the white lead, and then the mixture is ground with oil and formed into paint, sold under various names according to quality, thus: guaranteed white lead—firsts, seconds, thirds, and fourths, the proportion of white lead diminishing, and that of the adulterant increasing as we descend from the pure material. Guaranteed and best white lead is not pure, and does not mean pure white lead. Pure white lead can be purchased at some makers, but its price, if pure, can never fall below £19 or £20 per ton. To sophisticate white lead, and produce the various inferiors named, dry powdered white lead is needed as a starting point, and for this purpose principally arises the necessity for its production. If ground in oil the adulterants cannot be properly incorporated with it. Dry white lead is used for nothing else that could ever give rise to any great demand for it. We have already observed that the production of this dry and powdered white lead, is the most dangerous proceeding connected with this industry. Grinding in oil is unattended with any important consequence to the health and comfort of those employed. A serious drawback to the "stack" production, the china-like incrustation to which reference has already been made, is that it requires crushing, grinding, washing, and drying, and a second course of dry grinding after it is dried; the most objectionable step in its preparation.

Could the corrosion of the blue lead be effected in such a way as to prevent any discolouration of the material by the tan and wood—could the corrosion be so produced as to be easily separated from the buckle or grating on which it has been formed—could this separation be so effected as to prevent the

breaking up of the lead skeleton, and the presence of small pieces of metal in the detached crust of white lead, two principal reasons for washing and drying are removed. There is yet another consideration, that is the presence of acetate of lead, always found in varying quantities in the incrustation produced, and remaining at the close of the operation and conversion. To remove this, careful washing, and after stoving and drying, must be accomplished. The amount of this salt present is found to differ with each operation, and in various portions of the same make. The washing out of the acetate is never perfect, and it involves a large amount of labour.

Opinions differ as to the effect of this acetate if allowed to remain in the product. White lead makers on the "stack" principle aver that it should and must be washed out lest it should damage the qualities of the paint. This is questionable, and not one can produce practical evidence of its being the cause of any damage if still contained in white lead. Facts seem to deny its harmfulness in this respect inasmuch as the best prepared samples, those washed and dried, from the most careful makers, will be found upon analysis to contain more or less of acetate of lead. A large proportion of this salt in white lead may not be beneficial for many reasons, but a small percentage can do no harm, nay, for many purposes it may be good. There is no substance used for driers for white lead that is more esteemed than this acetate of lead, commonly known as "sugar of lead."

A small amount of this salt present in white lead would communicate drying properties, and this alone is what it could do. Granting that we can discover a method of producing white lead of amorphous character of good density, free from all discolouration, free from all particles of metallic lead, and free from but a small percentage of acetate of lead, then washing will not be needed. Stoving and drying become unnecessary. The work of women, their deadly occupation, so burdensome to the operatives, and to all with whom they are concerned, is done away with. Let us again call to remembrance that this work attendant upon washing and drying white lead cannot be pursued for any length of time, that lead colic, palsy, falling hands, and paralysis, the concomitants to breathing and handling white lead soon overtake the workers, that these symptoms are rapidly followed by premature decay and death. We surely shall hail with delight any method of remedying these grievous evils!

Such a process as that which I have characterised as being needful is invented, and it is my pleasing task to describe this plan to you this evening. It is Gardner's patent process for producing electric white lead. As the name implies, Professor Gardner calls electric power to his aid, and by its means good amorphous white lead can be produced in 14 days. No washing or drying are necessary. No women are engaged in the manufacture, and but few men. The white lead thus produced by the aid of electricity is deposited in a peculiar state of disintegration, it is perfectly amorphous and non-crystalline, of the purest quality, its density is 5.8. When ground in oil and made into paint it possesses great body and a covering power inferior to no other paint, if not superior to them all.

Painters who have used the paint, practical men, and amongst these we may observe coach painters, have pronounced its excellence and superiority to the best ordinary white lead paint.

By this electrical process of manufacture, not only is the time consumed in the making and in the preparation of this material greatly shortened, but the cost of preparation is reduced, and added to this is the important fact, the vital factor in our con-

sideration. The labour of women is unnecessary! No lives are sacrificed to its working requirements.

Professor E. V. Gardner, who has been for some years occupying his attention with the subject of white lead making, with a view especially to remedying its existing evils, has invented his electrical chamber process of manufacture, and an entirely new course of after treatment. He has for the last seven or eight years been more or less occupied perfecting his conception, and accommodating it to the practical and commercial claims.

Chamber processes are not new, there have been several patents enrolled for making white lead in closed chambers, but none have proved commercially convenient, or practically successful in their adaptations, and none have survived to the present time so far as I can ascertain.

In Germany white lead is made in chambers in the present day. The lead in gratings or sheets is supported on wooden rods, saddle fashion, the chamber is then filled and its contents are then submitted to currents of acetic acid vapour, air, steam, and carbonic acid gas; the time needful for conversion is six or seven weeks. The after steps in the separation of the incrustation, and its preparation for trade purposes are much the same as in the "stack" product preparation. It is washed, stoved, dried, and ground. The white lead made on the German plan does not differ in any material degree, save it be in price, from the best English commodity. We may assume that the washing and drying in Germany consumes a like period of time to that process in English works, viz: two or three weeks. We then see that the German plan of making white lead cannot be perfected in less than eight or ten weeks time from the commencement of the corroding action in the chamber. Let us compare these facts and the efficiency of the different plans I have described.

The time required to complete the corrosion in the stack is at least 14 or 16 weeks.

The Gardner's electric process requires for the same purpose only 14 days. As to point of time the German plan excels the stack, and can be carried out in one half the time required in the stack method of conversion. The Gardner's electrical method excels the German, and can be perfected in one-third the time needed for the German chamber operation, and one-sixth the time required by the stack.

These figures open out a most important matter when we regard the capital invested and lying dormant in stack lead works.

We will particularise this Gardner's electrical plan of working.

It is well known that we have in electricity a most powerful agent by which to effect the chemical combination of various substances on the one hand, or on the other, by its means to break up and disrupt a chemical compound. Professor Gardner's main principle of action in his new process to which we now refer is founded upon these facts, and he takes advantage of electrical power to cause the combination of the lead with the necessary elements to build up white lead in his chambers. He either employs electrical discharges to energise and render active in their chemical affinities the various materials engaged, or he so disposes of them as to form an electric galvanic combination in the chamber. In the latter arrangement the chamber and its contents represent a gas battery on an extensive scale. In practice he prefers the latter plan; it is more simple, more manageable in the hands of the ordinary workmen. The original plan was to have graphite or graphitic carbon plates or supports for the buckles or gratings of lead within the converting chamber. These carbon plates and the lead to be converted,



were so placed as to form a collection of galvanic "couples" or "pairs," and in this condition were submitted to the gases entering the chamber from without. It will be understood that various modes of arrangement would occur without departing from the principle concerned.

In practice this method answered very well, but presently a difficulty arose; not only were the graphitic carbon or graphitic plates expensive, but they were easily broken, and became friable in use. Carbon plates of an especial kind were manufactured to meet these failures and remedy these defects, not without success, but still open to objection. In looking round for some substitute to replace the carbon two points were to be kept in view, to seek some electro-negative to lead like unto the carbon, and some electro-conductive like unto it, and some material that would bear rough handling, such as workmen give, and be practically convenient in its adaptation to a working chamber.

Gold or platinum would excel in these particulars, but there are considerations which debar their use. Pure tin presented itself and tin was tried with great success. Tin would at first be thought, on account of its close electrical relationship to lead, far from favourable to the purpose. Graphite or carbon would appear far more suitable. Practice pronounced the tin to act as efficiently as carbon. This may at first seem contradictory and strange, but if we consider that while the carbon is certainly more highly electro-negative to lead than tin, yet the tin is the more conductive, and offers the less resistance to the electric current of the two, in this manner the tin compensates by its conductive power all it may lack, as compared with carbon, in electrical energy when coupled up with lead.

Tin plate is now used as the electric-negative element in the chamber of the Gardnerplan. Tin plate means pure tin, Ingot tin is rolled out into plates, the bottom of the chamber is covered with this pure tin plate, so are the bearers and the shelves, or supports which are to hold the lead during its conversion. Tin pipes and tin fittings which resist the action of acetic acid, are also used to conduct the gases to the chamber to carry on the converting operation, and to preserve the product from any source of discolouration.

When a chamber is prepared for the converting operation, the whole of the lead it contains will be in metallic communication with the tin supports, and these with the tin covered bottom of the chamber.

The chamber when working is kept at a certain temperature by a steam coil beneath the floor of the chamber. The process is simple. The lead buckles or gratings are placed on tin covered stands, somewhat in form and make like unto a dinner-wagon. The whole is hauled up and dipped into a bath of acetic acid and acetate of lead; it there remains for one or two minutes, it is then hauled out, drained and lifted bodily through the top into the chamber. Other stands filled with buckles are so dipped and so placed till the space of the chamber is fully occupied.

This dipping cleanses the surface of the metal, and when it is exposed to the air it is speedily coated with a hydrated oxide of lead. This is the first step in the process of conversion. The chamber when filled is closed, and its temperature brought to about a 100° Fahr., then vapour of acetic acid and vapour of water and air are supplied from without to the interior of the chamber. This is continued for 15 or 20 hours. The lead buckles within the chamber will now possess a whitish coating consisting of subhydrate and subacetate of lead, and they will present a uniform colour. Carbonic acid generated in any convenient manner is next passed

into the acid generator, it mixes with the other gases and vapours, and with them goes on its way to supply the chamber. Speedily the action of the carbonic acid is observed, the surface lead becomes quite white and presents the appearance of a snow shower having fallen within the chamber. The formation of white lead is now speedily effected. This treatment is continued throughout the space of 13 days; at the end of this time the supply of acetic acid vapour is stopped, and the supply of air, steam and carbonic acid continued, according as it is desired to obtain white lead rich in oxide or in carbonate.

After a short further period steam and air only are sent into the chamber, which is varied in temperature to 120° or 130° Fahr., and lastly the steam supply is stopped, air alone enters the chamber, which is kept heated by the coils beneath the floor. The contents of the chamber are now in a dry state, and the operation is terminated.

It will occur to most of us that these terminal proceedings amount in effect to a convenient method of washing and drying the white lead while it is still attached to the parent lead, and this it is in fact.

The contents of the converting chamber are lifted out through the opened top, and the buckles or gratings with their crust of white lead are turned into the agitator. This agitator is an iron cage revolving inside a closed chamber of the same material. During the revolution of this cylinder or cage the contained lead gratings fall from side to side, and the incrustation on their surfaces becomes detached and broken up. It falls in this broken state through the bars of the cage or cylinder into a receptacle beneath. The denuded buckles or gratings are retained in the cylinder and are removed. These gratings or buckles are cast of such a thickness as to withstand two or three converting operations in the chamber before they are recast.

This crude white lead is carried by an elevator, or it falls into the hopper of a pair of granite crushing rolls, also enclosed, and from these it passes into the mixer or incorporator from which it can be removed in a dry state or mixed with oil.

The incrustation of white lead will be found upon examination to be possessed of some peculiarities, the result of the electrical action which has been going on within the chamber. It is quite white; it falls from the lead buckle or grating which it coats; if the grating be struck against a piece of wood, with but a slight blow, it is easily friable, and can be rubbed to the finest powder between the thumb and finger, or on the palm of the hand.

Now, we may explain as we conceive the philosophy of its production in this state of disintegration.

We know that a feeble and prolonged current of electricity will in time deposit metals from their solutions in a crystalline condition, and that if we quicken the current of electricity and cause it more energetically to act on the same solution, we can precipitate the metal from that solution in a state of powder.

It is to similar action of electricity as that to which we last refer that we ascribe the formation of the crust on the gratings of lead in the non-adhered and disintegrated condition in which it is produced, and by reason of which it is so easily detached from the lead and broken up to powder. No edge runner grinding, such as is required by the stack process, is in this case necessary.

The crude white lead and crushed material, whether in a dry state, or incorporated with oil, is finished and ground in a granite roller paint mill, from which it issues as dry white lead, or as white lead in oil.

Paint made from this electric white lead has been sent to America, to France, to Belgium, to Germany

for trial, and has also been largely tested in this country by painters, engineers, and others unacquainted with its precise nature, and it has been productive of good results.

Of its density, body and covering point, there can be no doubt, and never once have these qualities been called in question.

The cost of the manufacture of white lead by the stack process is about £3 to £3 10s. per ton, by the German method the cost is about the same as by the stack. By Gardner's electric process the cost of conversion is 10s. a ton. To this 10s. must be added the cost of labour expended in preparation, an item which cannot be well estimated on the present limited scale of manufacture; it could not exceed an additional 15s. a ton. This would bring the cost of manufacture of electric white lead to 25s. a ton.

In this electric process inferior lead can be operated upon with success. Brands of that metal, such as white lead makers, by the stack method, dare not employ, may be successfully converted in an electric chamber, and with fair results as to the quantity and quality of the white lead produced.

By the use of the Gardner's electric process it would appear that we not only preserve health but save lives; we not only save time but interest on large capitals, which lie idle for long periods at a time, but we can economize and simplify the whole manufacture and preparation of white lead, divesting it of all its present cumbersome and unhealthy stages. Gardner's process, we believe, must take a prominent position as one of the most necessary, valuable, and scientific inventions of modern times.

#### ON JAPANESE CAMPHOR—ITS PREPARATION, EXPERIMENTS AND ANALYSES OF THE CAMPHOR OIL.

BY H. OISHI.

(COMMUNICATED BY T. TAKAMATSA.)

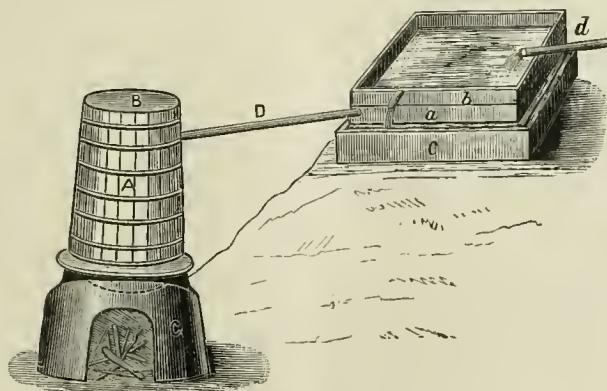
*LAURUS CAMPHORA*, or Kusunoki, as it is called in Japan, grows mainly in those provinces in the islands Shikoku and Kinshin, which have the southern sea coast. It also grows abundantly in the province of Kishu.

The amount of camphor varies according to the age of the tree. That of a hundred years old is tolerably

flange at the top. Over this flange a wooden tub A is placed, which is somewhat narrowed at the top, being 1ft. 6in. in the upper, and 2ft. 10in. in the lower diameter, and 4ft. in height. The tub has a false bottom for the passage of steam from the boiler beneath. The upper part of the tub is connected with a condensing apparatus by means of a wooden or bamboo pipe D. The condenser is a flat rectangular wooden vessel (a), which is surrounded with another one (c) containing cold water. Over (a) is placed still another trough (b) of the same dimensions, into which water is supplied to cool the vessel (a) at the top. After the trough (b) has been filled with water, the latter flows into (c) by means of a small pipe attached to it, as shewn in the figure. In order to expose a large surface to the vapours, the condensing trough (a) is fitted internally with a number of vertical partitions, which are open at alternate ends, so that the vapours may travel along the partitions in the trough from one end to the other. The boiler is filled with water, and 120kg. of chopped pieces of the wood are introduced into the tub, which is then closed with a cover, cemented with clay, so as to make it air-tight. Firing is then begun; the steam passes into the tub, and thus carries the vapours of camphor and oil into the condenser, in which the camphor solidifies, and is mixed with the oil and condensed water. After twenty-four hours the charge is taken out from the tub, and new pieces of the wood are introduced, and distillation is conducted as before. The water in the boiler must be supplied from time to time. The exhausted wood is dried and used as fuel. The camphor and oil accumulated in the trough are taken out in five or ten days, and they are separated from each other by filtration. The yield of the camphor and oil varies greatly in different seasons. Thus much more solid camphor is obtained in winter than in summer, while the reverse is the case with the oil. In summer from 120kg. of the wood, 2.4kg., or 2% of the solid camphor are obtained in one day, whilst in winter, from the same amount of the wood, 3kg., or 2.5% of camphor are obtainable at the same time.

The amount of the oil obtained in ten days, *i.e.*, from 10 charges or 1200kg. of the wood in summer is about 18 litres, while in winter it amounts only to 5.7 litres. The price of the solid camphor is at present about 1s. 1d. per kilo.

The oil contains a considerable amount of camphor



rich in camphor. In order to extract the camphor, such a tree is selected; the trunk and large stems are cut into small pieces, and subjected to distillation with steam.

An iron boiler of 3ft. in diameter is placed over a small furnace C. The boiler is provided with an iron

in solution, which is separated by a simple distillation and cooling. By this means about 20% of the camphor can be obtained from the oil. The author subjected the original oil to fractional distillation, and examined different fractions separately. That part of the oil which distilled between 180°-



185° C, was analysed after repeated distillations. The following is the result :—

	Found	Calculated as $C_{10}H_{12}O$ .
C	= 78.87.....	78.95
H	= 10.73.....	10.52
O	= 10.40 (by difference) .....	10.52

The composition thus nearly agrees with that of the ordinary camphor.

The fraction between 178°-180° C, after three distillations, gave the following analytical result :—

C	= 86.95
H	= 12.28
	99.23

It appears from this result that the body is a hydrocarbon. The vapour density was then determined by V. Meyer's apparatus, and was found to be 5.7 (air=1). The molecular weight of the compound is therefore  $5.7 \times 14.42 \times 2 = 164.4$ , which gives

$$H = \frac{164.4 \times 12.28}{100} = 20.18$$

or  $C_{12}H_{20}$

$$C = \frac{164.4 \times 86.95}{100} = 11.81$$

Hence it is a hydrocarbon of the terpene-series, having the general formula  $C_nH_{2n-4}$ . From the above experiments, it seems to be probable that the camphor oil is a complicated mixture, consisting of hydrocarbons of terpene-series, oxyhydrocarbons isomeric with camphor, and other oxidized hydrocarbons.

#### APPLICATION OF THE CAMPHOR OIL.

THE distinguishing property of the camphor oil, that it dissolves many resins, and mixes with drying oils, finds its application for the preparation of varnish. The author has succeeded in preparing various varnishes with the camphor oil, mixed with different resins and oils. Lampblack was also prepared by the author, by subjecting the camphor oil to incomplete combustion. In this way from 100c.c. of the oil, about 13grms. of soot of a good quality were obtained. Soot or lampblack is a very important material in Japan for making inks, paints, &c. If the manufacture of lampblack from the cheap camphor oil is conducted on a large scale, it would no doubt be profitable. The following is the report on the amount of the annual production of camphor in the province of Tosa up to 1880 :—

	Amount of Camphor produced.	Total Cost.
1877 .....	504,000 kins .....	65,520 yen
1878 .....	519,000 " .....	72,660 "
1879 .....	292,890 " .....	74,481 "
1880 .....	192,837 " .....	58,302 "

(1 yen=2s. 9d.)  
(1 kin=11lb.)

#### A FEW REMARKS ON PARAXYLENE.

BY IVAN LEVINSTEIN.

A FEW months ago I communicated to the Manchester section a method for determining the commercial value of xylene (*Journ. Soc. Chem. Ind.*, Feb. 29, 1884, page 77). I then stated that of the three existing isomeric xylenes metaxylene alone has thus far been applied to any special technical use, and my method was principally directed towards the determination of the relative amount of this substance present in crude xylene. I also referred in the same communication to a method for estimating the approximate amount of paraxylene present in crude xylene, and it is in reference to this method, which I had based on O. Jacobsen's investigations, that I am induced to

trouble you with a few remarks, though I am afraid that the subject will not interest many of you. In the *Journal of the Chemical Society* for April, 1884, Messrs. Armstrong and Miller state in the footnote, on page 151, that Jacobsen is wrong in remarking that "on mixing crude tar xylene (Ber 10.1009) with ordinary sulphuric acid, even when warm, only small traces of paraxylene are dissolved," as, in their opinion, paraxylene may be dissolved by well agitating it with ordinary sulphuric acid almost as easily as pure metaxylene. On reading this communication it occurred to me that Armstrong and Miller's observation relative to the solubility of paraxylene can only apply to that substance in a pure unmixed state, whilst Jacobsen is distinctly referring to the solubility of paraxylene when present in crude tar xylene. That being so, I fail to see where Jacobsen is really wrong, but if Messrs. Armstrong and Miller contend that paraxylene when mixed with paraffins and other hydrocarbons, which always are present in crude tar xylene, dissolves almost as easily as pure metaxylene, then I think that their statement is incorrect, as the result of the following experiments will show :—

I first ascertained the solubility in ordinary sulphuric acid of pure paraxylene and pure metaxylene, when it was found that about 150c.c. of acid are required to dissolve 100c.c. of paraxylene, while only about 125c.c. of acid are necessary to dissolve the same quantity of metaxylene. Thus it was shewn that as regards the action of sulphuric acid on either pure para- or pure metaxylene there was no great difference in the solubility of either, a fact which I have good reason to believe was also well known to Jacobsen. I then prepared a mixture of 100c.c. paraffins (as present in crude xylene), 100c.c. pure paraxylene, 150c.c. ordinary sulphuric acid (a quantity just sufficient to dissolve the 100c.c. paraxylene). This mixture was continuously agitated for half an hour, and afterwards it was well mixed for about 30 hours altogether, when only 60c.c. of paraxylene were found to be dissolved, showing that in the presence of paraffins, the paraxylene did not dissolve nearly so readily as when treated in a pure unmixed state. I then varied the experiment by making a mixture of 80c.c. metaxylene, 10c.c. paraxylene, 10c.c. paraffins, 150c.c. ordinary sulphuric acid. This mixture was well agitated for about half an hour, when 15c.c. of the hydrocarbons remained undissolved, which, on examination, proved to be composed of 10c.c. of paraffin and 5c.c. of paraxylene. The same experiment was repeated, the agitation of the mixture being continued for about five hours, when all the paraxylene at last dissolved. This experiment proves that paraxylene when mixed with paraffin and metaxylene is not so easily acted upon by ordinary sulphuric acid as metaxylene.

I finally varied the experiment by using the foregoing proportion in the mixture of the hydrocarbons, but reducing the amount of sulphuric acid, viz. :—80c.c. metaxylene, 10c.c. paraxylene, 10c.c. paraffin, 100c.c.  $H_2SO_4$ . After agitating for several hours, 20c.c. of hydrocarbons remained undissolved; these 20c.c. were carefully analysed, and consisted, as nearly as possible, of 10c.c. paraffins and 10c.c. paraxylene. In this instance there was a complete separation of the para- and metaxylene. These experiments prove that paraxylene does not "dissolve almost as easily as metaxylene" when it is present in crude tar xylene, which always contains more or less paraffins, and that the solubility of paraxylene in ordinary sulphuric acid depends (1) on the purity of the paraxylene; (2) on the quantity of sulphuric acid employed; (3) on the time during which the agitation of the acid with the paraxylene is con-

tinued, and I must still adhere to the opinion expressed in my previous communication, that the difference in the solubility in sulphuric acid of paraxylene and metaxylene when present in crude gas tar xylene is sufficiently great to make it practically applicable to a method for separating and approximately determining the amount of paraxylene present in crude xylene.

## NOTES ON THE CONCENTRATION OF SULPHURIC ACID.

BY PETER HART.

THE concentration of sulphuric acid economically seems to have been a problem for a long time, if one may judge by the many methods proposed. I doubt my ability to mention them all, or even to give a proper chronological sequence to those which I can call to mind. The late Mr. Hannibal Becker once described to me the method used at his father's works early in this century. Comparatively small retorts of the ordinary beaked type were employed these were set in sandbaths and, the boiling completed, were cooled; removed, in their arms, by men wearing sheepskin aprons; emptied, and refilled to go again through the same round of operations. Platinum, until recently, was not largely employed, perhaps the great first cost stood much in its way. Messrs. Tennants and Co. of this city had one in use from 1852 to about 1864, when it was sold for scrap, and replaced by the large glass retorts at present employed. Platinum, though generally supposed to be unacted upon by sulphuric acid, does disappear when used for concentration. Well do I remember the latter portion of this still's life; the constantly-recurring Monday morning's work of repairing, the inserting my head into its mouth to find the little pinholes, the marking them, and then, by means of the blowpipe, the soldering them up, using fragments of a half-sovereign, until, in the fulness of time, it seemed to have broken out into a rash of gold spots all over. During 1857 the late Mr. Gossage was experimenting with an apparatus, which, in fact, was simply an anticipation of the present Glover tower, filled with flints or silicious pebbles, where a descending current of chamber or brown acid was met by an ascending current of hot air, this latter eventually substituted by the hot gases from a coke fire, but, whether from its being impracticable or because he did not work sufficiently long at it, he was not successful. Since that time this same idea has cropped up many times, of course more or less modified. It will suffice to mention only the proposals to concentrate in vacuo, the blowing air through at comparatively low temperature, say 300° Fahr. As to the constant flow systems, with glass, earthenware, or platinum, a great number have been proposed, one of which, at least, is, or was, working successfully at Messrs. Chance's at Oldbury, and other places. None of these seem to have been so strikingly good as to have made much way in the trade, or to have been so generally adopted as to indicate a thorough success. The most startling proposition is to concentrate in iron. The first to propose this, to my knowledge, is Hartmann, No. 2839, A.D. 1879, 11th July, who proposes employing iron vessels for this purpose. He says "iron vessels are not available for evaporating the acid in the state in which it is drawn from the leaden evaporating vessels, as the acid in this state acts upon iron energetically," but he has discovered that "iron vessels

may be employed in the final concentration with impunity and success if the acid be first fully charged with sulphate iron. This sulphate iron does not remain in the acid as a permanent impurity, but as the strength of the acid increases during the evaporation the iron becomes insoluble, until but a trace is left in the acid." The remainder of the specification is taken up with details of working which anyone may obtain from that source. Alfred Julius Boulton, for James Gridley, of Brooklyn, New York, No. 4709, 3rd Oct., 1882, received provisional specification for his invention. He says: "My invention is based upon the fact that sulphuric acid of 66° Beaumé at 60° Fahr. has little or no action on cast iron, and my invention consists first of the process of introducing a small stream of dilute acid from the evaporating pans, of strength, say of 60° Beaumé, into a large quantity (say one ton or more of acid of the strength of 66° Beaumé) contained in a concentrating pan or retort charged originally with acid of 66° Beaumé, and kept there after at the boiling point. The feed of dilute acid to be so regulated as not to reduce the acid in the concentrating pan or retort below 65° Beaumé at the surface or acid line, at which point, and four inches below the surface, the pan at the sides is protected from the action of the acid." These seem to be the guiding principles of this invention, the remaining portion of his paper being confined to descriptions of apparatus.

Mr. Menzies, Patent No. 3230, 29th January, 1883, proceeds to attack the arsenic problem, a point overlooked or intentionally neglected by the previous inventors. He moves much along the same lines as regards strength, etc., as the others just mentioned. He oxidises the arsenic and iron to the highest point, by means of nitrous vitriol, nitric acid, or nitrates, boils off the remaining water, and then proceeds to distill off the remaining strong acid, using glass tubes as a condenser, thus obtaining a pure acid, as all the impurities remain in the still to be cleaned out periodically. It seems surprising that technical chemists should, for so many years, have tortured their brains devising every possible configuration of glass or precious metal, and have overlooked the fact that common prosaic cast iron could be employed. This is the more remarkable as it has been known for at least thirty years, that cast iron was but slightly acted upon by boiling sulphuric acid. It will be known to many of you that an operation known as "parting" takes place at the Mint and other places. This "parting" means the separating silver from gold. The granulated alloy, or mixture of these two metals, was, at one time, treated with nitric acid, to dissolve out the silver, eventually sulphuric acid was substituted, using platinum boilers, but in Phillips *Metallurgy*, published in 1852, it says:—"The granulated mixture (meaning alloy) is now placed in large cast-iron boilers, into which are thrown two and a half times its weight of strong sulphuric acid, of s. g. 1.848 (169 Tw.), and the whole is at once heated to ebullition by a fire placed beneath the pans." The remainder of the process need not detain us. The point of interest lies in the fact that here the germ of concentration in iron has remained hidden for such a length of time. I do not claim for these somewhat misty and straggling notes any value either historical or practical; if they point a moral at all it is, that in general scientific and technical literature, there exist the buds of many serviceable inventions, and that, in these latter evil days of much work for small reward on which we have fallen, it behoves us all to look around and exercise the whole of our skill and talent to reduce the cost of production to the utmost, for however politicians and students of the gloomy science may squabble, this is true protection of native industry.



## REPORT UPON STANDARD METHODS OF SAMPLING, ANALYSIS, AND STATING THE RESULTS.

YOUR Committee, as instructed by the resolution unanimously agreed to at the Conference of April 1st, has carefully considered the remarks made at that meeting, and also the opinions expressed in the other Sections of the Society.

The resolution of April 1st, proposing "National" methods as against "International" ones, represents the very strong feeling which exists upon the subject in this Section, and your Committee is unanimously of opinion that a national agreement, as far as regards sampling and stating the results, would meet all purposes.

With regard to the question concerning uniform methods of sampling, your Committee is of opinion that this matter could be satisfactorily arranged by appointing sub-committees of the various branches of trade in which reports of analysis are required, and that such sub-committees should determine the manner in which samples should be taken. Each sub-committee should deal with the special articles in which its members are interested and experienced, and should report to a General Committee, which may be appointed to consider the subject.

After carefully reconsidering the report and the various discussions which have taken place in the different Sections, your Committee is unanimously of opinion that it would be practically impossible to insist on certain standard methods of analysis, and that chemists may be left to use the most reliable. Your Committee, however, recognises the fact that in many cases it would be most desirable that chemists should state the method used upon the certificate of the analysis or assay.

As to uniform methods of stating the results of analyses or assays, your Committee is of opinion that such an agreement would be possible, but as only the members of the various trades, or their scientific advisers, would be likely to know the requirements of those trades, your Committee suggests that the question of stating the results should be discussed by the sub-committees having charge of the question relating to uniform methods of sampling.

As to the appointment of a general committee and its powers, your Committee is of opinion that such a question may safely be left to the Council and a general meeting of the Society; believing, however, that the question can only be satisfactorily dealt with by a large committee, in which the many branches of trade are adequately represented.

Your Committee is unanimously of opinion that any agreement which may be suggested should only be considered as a recommendation to chemists, buyers, sellers and consumers, and that no attempt should be made to make such methods absolutely binding on the parties concerned, though your Committee feels sure that the publication of the best methods in the Journal of the Society would, in a short time, have considerable influence in securing their adoption.

Your Committee would also suggest that any standard methods should be revised by the general committee from time to time.

This report was received unanimously, and passed, with the request that it be forwarded to the Council.

### CLOSING OF THE SESSION, 1883-4. THE CHAIRMAN'S ADDRESS.

The current session closes with the meeting of this evening, and the first meeting of the new session will take place in October. In taking a retrospect of the

work which has been done by the Manchester Section during the past twelve months, we may well feel proud of the result of its labours. Not only have we had a large number of important and interesting papers, but the constantly increasing number of gentlemen attending our monthly meetings proves that our work has been appreciated by many who are not yet actual members of our Society. The attendance at our meetings has averaged over 300, while almost every month has brought us from our own district, from ten to fifteen new members, so that our Section is already represented by the somewhat formidable figure of about 350; which figure, however, I hope may be doubled before the close of the next session. A number of the papers read during the past twelve months had especial reference to the important industries of this district; and it is worthy of note that our meetings were generally best attended when the subject of the paper had a bearing on dyeing, printing or bleaching, or some of the industries more directly connected with them. I think this fact ought not to be lost sight of in the future management of our Section, and in my opinion each individual Section can render the best service in advancing the objects of the Society, by reading and discussing papers on subjects which have, more or less, a special bearing on the principal industries located in its own district. Some of those present may perhaps be aware that a new society has been formed at Bradford, under the name of "The Society of Dyers and Colourists." This society was formally opened last month in an inaugural address by Dr. Roscoe, and it already numbers 150 members. When it became known that it was in contemplation at Bradford to start a society of dyers and textile colourists, many of us felt desirous that our Yorkshire friends might see their way to amalgamate with our Society, and to form a special section, and I may say this was also my desire. Our friends could not, however, be induced to alter the determination they had come to of founding a separate society of their own; and now that this society has been actually brought into existence and established for an object partly identical with ours, viz., that of advancing and promoting scientific and technical knowledge, it is only becoming in us to wish it every success. Some of our members thought that the Manchester Section had already made a speciality of the subjects of dyeing and printing, that there was really no room for a new society, and that such an association as that contemplated might even draw away some of our members. I beg, however, to differ from this latter opinion, and I think that so far as the effect on our members is concerned, the new Society will, on the contrary, add to the number of members of our section, and already three gentlemen have joined us from the Society of Dyers and Colourists. We must also not forget that Bradford is a most important dyeing centre, and that dyers from that district cannot conveniently attend our meetings regularly. But there is another and more weighty reason why the new Society, if properly managed, should become a very desirable and useful one. With all due respect to the practical skill, industry and sagacity of our dyers, they are not as a rule chemical *savants*, and the majority of our papers and of the other valuable communications to be found in our excellent Journal, pre-suppose for their proper comprehension a standard of knowledge superior to that of most of those gentlemen. The principal work of the Society of Dyers and Colourists will, therefore, consist in endeavouring to advance technical and scientific knowledge amongst this class of men, who have not had the opportunity of passing through any regular scientific training, or of acquiring that accurate scientific knowledge which is so useful and valuable

in their different trades. The society in question will, therefore, not be a rival to our own, even though we are both pursuing to a great extent the common object of advancing technical and scientific knowledge; but will, on the contrary, be of great assistance in preparing the ground, and thus materially aiding the very purpose with which our own Society has been founded. On the other hand, there are many dyers, especially of the younger generation, who have acquired a fair amount of technical knowledge, and it is these to whom our section must direct its attention, and whom we must show by our labours that it will be to their benefit to join our Society. It will, therefore, be to our own interest and the interest of all who really have at heart the diffusion and advancement of technical and scientific knowledge, to co-operate in a friendly manner with the Bradford Society of Dyers and Colourists. I have great pleasure in informing you that the council of the new society has already passed a resolution allowing any member of our Manchester Section to attend their ordinary monthly meetings, and fixing the dates of those meetings so as not to clash with the dates at which our meetings are usually held. I doubt not, gentlemen, that you will gladly reciprocate this friendly action on their part by permitting members of the new society to attend our ordinary monthly meetings. It is by thus harmoniously working together, and affording each other mutual support and recognition, that societies founded with objects such as ours may best attain their ends, and confer the greatest benefit on their members and the world at large.

## Glasgow and West of Scotland Section.

*Chairman:* Ed. C. C. Stanford.

*Vice-chairman:* Professor Ferguson.

### Committee:

George Beilby.  
Professor Crum Brown.  
J. Christie.  
Dr. John Clark.  
Professor Dittmar.  
George G. Henderson.  
George M'Roberts.  
James Maclear.

Professor Mills.  
James Napier, jun.  
T. L. Patterson.  
J. B. Readman.  
Dr. Edmund Ronalds.  
F. J. Rowan.  
R. R. Tatlock.  
Dr. William Wallace.

### Hon. Treasurer:

J. J. Coleman, 45, West Nile Street, Glasgow.

### Hon. Secretary:

Dr. James J. Dobbie, Chemical Laboratory, University of Glasgow.

### ANNUAL GENERAL MEETING.

Immediately after the Ordinary Meeting of the 13th May, the Annual General Meeting of the Section was held, at which, the Chairman, Vice-Chairman, Secretary, Treasurer, and four members of committee, who had been balloted to retire, were all re-elected for another year of office.

*A Meeting was held in the Rooms, 207, Bath Street, on Tuesday, May 13, 1884.*

MR. E. C. C. STANFORD IN THE CHAIR.

## ON DIFFERENT METHODS OF PRODUCING COLD ARTIFICIALLY.

BY J. J. COLEMAN, F.I.C., F.C.S.

An essential constituent of any machine hitherto used for producing cold continuously, is an elastic fluid or vapour, which may be atmospheric air, ammoniacal

gas, sulphurous acid gas, ether vapour, methylic ether vapour, &c., &c. Such vapour is alternately compressed and expanded, during which certain phenomena manifest themselves, and a succession of such constitutes the working of a machine for producing cold. If these cycles be studied, it will be found that they all involve introducing energy [in the form of heat, or its equivalent mechanical work] into the cycle, and *then rejecting it*; by this means the vapour is brought into that physically condensed condition, by which it will absorb heat from an external substance by its own expansion or evaporation. A cold producing machine abstracts heat from the substance being cooled by the spontaneous expansion or evaporation of an elastic fluid inside the machine, and to keep up a succession of such expansions and abstractions of heat from an outside body, heat or energy has to be given to the machines.

First let us take the production of cold by atmospheric air. In this case we have four distinct stages in the cycle, the first of which is that air is compressed by mechanical force or work, which not only compresses the air, but makes it exceedingly hot, for according to the laws of thermodynamics 772 foot pounds of energy used in compressing the air appears as a unit of heat in the compressed air itself. The second stage of the cycle is therefore to get rid of this heat actually put into the air, and this is done by injecting into the air, well, river, or sea water, or by passing the compressed air through tubes surrounded with such water. This brings us to the third stage of the cycle in which we have compressed air of *atmospheric temperature*. If we then make this compressed air work an engine constructed like a steam engine, it develops force and becomes immensely cold; and for every 772 foot pounds of mechanical energy developed, there is a reduction in temperature equivalent to one heat unit. We have now arrived at the fourth stage, which is, that the cold air is brought into contact with the substance we wish to cool, and goes back for another cycle. There is another way of producing cold by atmospheric air, known as the vacuum method, and it consists of three stages. In this case mechanical force is used to lift the piston of an air pump, the energy here being rejected in the friction or heat of displacing the atmosphere. The rarefied air inside the pump is made to pick up heat from the substance being cooled, and being restored to its normal pressure is expelled. When ether is used the cycle is as follows: By mechanical work the ether vapour is pumped from the reservoir of ether and then passed in a compressed state through pipes surrounded with cold water; here the energy introduced by the pump is rejected in the form of heat, which is carried away in the cooling water; the liquid ether then passes on into a reservoir, and then evaporates into the vacuum maintained by the pump; in fact, the ether evaporates so rapidly in vacuo that it boils at a temperature approaching zero Fahr., and the liquid ether consequently abstracts heat from any substance we wish to be cooled by the machine. The cycle in the case of Pictet's Sulphurous Acid Gas Machine is precisely the same, only the whole apparatus is worked at higher pressure, owing to the boiling point of liquid sulphurous acid being lower.

Machines have also been introduced by Prof. Linde, in which anhydrous ammonia is used instead of sulphurous acid gas; that is, ammonia gas absolutely free from water compressed by the mechanical force of a pump; the energy thus introduced is abstracted by passing the hot gas through pipes surrounded by water, and the liquid ammonia is then allowed to boil in vacuo created by the pump, which is at a temperature still further below zero, than in the case of the sulphurous acid or ether, and thus abstracts



heat from the brine or other liquid being cooled. The most common form, however, of the ammonia machine for producing cold is that introduced by Carré, and known as the Ammonia Absorption Machine, and which has been much improved by Reece and others in this country. The cycle is very interesting to study. Ordinary very strong commercial liquid ammonia is put into a boiler of iron connected with an upright tower of shelves, a fire is placed under the boiler, and the mixed ammonia gas and steam ascend the tower; the steam condenses and drops back as water into the boiler, whilst the ammonia gas goes out of the top of the tower, and then descends through a coil arranged in a second tower filled with cold water; this cold water, of course, condenses the ammonia gas inside the tubes, which by this time has got, not only very hot, but very much compressed, there being a valve at the outlet of the coil which prevents the free flow of the gas, until by its accumulated pressure it liquefies itself. Comparing now this with the first two stages of the compressed air cycle, we have repetition of the same phenomena. We impart energy to the gaseous ammonia, we abstract it again in the water cooling the coil of pipe. The product liquid anhydrous ammonia then goes to the next stage where it is allowed to evaporate or expand spontaneously by release of pressure.

This evaporation is usually accomplished in a vessel containing pipes through which brine or other liquid to be cooled circulates. We now arrive at the fourth stage of the process in which we have simply ammonia gas at atmospheric pressure, and in order to complete the cycle we have to make this gas come into contact again with water, and so become absorbed, and this is done in a fourth vessel in which heat is rejected, after which the reformed ammonia solution goes back to the boiler from which it started.

Another class of cold-producing machine was devised by myself in the year 1876, and has been used on the large scale by Young's Paraffin Light and Mineral Oil Company for condensing volatile liquid hydrocarbons existing in the waste illuminating gas produced in distilling shale for oil. In this process energy was employed in compressing the hydrocarbon gases to a pressure of about 150 lb. to the inch, this energy was rejected by passing the compressed gases through a system of tubes, surrounded by cold water, a number of liquid hydrocarbons being thus obtained. The compressed gas was then passed through a second system of pipes, and then expanded in a cylinder, giving mechanical power to the crank shaft working the machine, the cold expanded gas being made to circulate round the second system of pipes, gave a second crop of liquid hydrocarbons (the product of joint cold and pressure), and consisting chiefly of amylene and other olefines, after which the gas was burnt as fuel.

Reverting to the definition with which I started, viz.; that all cold-producing machines involve introducing energy in the form of heat or mechanical work, and then rejecting the same before the vapour is brought into that physical condition that it will expand spontaneously each cycle: we may extend the definition, and state that spontaneous expansion involves the lifting of heat or energy from the substance being cooled and transferring it (either as heat or mechanical work) to another body, and the extent to which this can be done in each cycle depends upon the energy introduced and then rejected in another part of the cycle.

A short time ago I had the honour of bringing this subject before the Institute of Civil Engineers in London, and in closing the discussion Sir William

Armstrong made some remarks which will now be easy to understand. He said—"To the uninitiated a compressed air cold producing machine did appear to form an exception to the general rule that all mechanical energy is ultimately converted into heat, for it resulted in the production of an opposite condition, but looking a little more closely into the matter it would be perceived that the machine was really a heat-producing machine, and that cold was merely the result of the fact that this heat was abstracted from the air, which thereby became a *medium of refrigeration*."

As atmospheric air is practically perfectly elastic, there is practically no limit to putting energy into it, and then removing the same, thereby increasing its density; consequently there is no practical limit to the cold capable of being produced in each cycle by the compressed air, but any machine which is worked through the medium of a readily condensable vapour, such as ammonia, ether, methylic ether, or sulphurous acid, has its action limited by the boiling point of the volatile liquid: it is therefore impossible with such machines to get so large a range of cooling in one operation as can be accomplished by air. The low temperatures which Pictet required for the liquefaction of oxygen and hydrogen were obtained in stages, first by ebullition of liquid sulphurous anhydride in vacuo producing sufficient cold to liquefy carbonic acid gas at a pressure of four atmospheres, and then in taking advantage of the still greater cold produced by the ebullition of the liquid carbonic acid gas in vacuo. There is, however, no reason to suppose that the same or much lower temperatures could not be obtained by the compression and expansion of air in a single operation.

The boiling point of ether under ordinary atmospheric pressure is 95° Fahrenheit, so that in order to use it as a medium for refrigeration it requires to be evaporated in vacuo—that is, pumping is needed, which causes it to boil rapidly, and it becomes cooled as the vacuum is increased; but the cooler it becomes the more slowly it evaporates, until, when its temperature sinks to a little below zero, evaporation ceases altogether, although the pump may be maintaining the vacuum. It follows from this that if the brine, which is usually the medium being cooled, returns back to the boiling ether without having picked up heat from the substance being cooled, the action is gradually diminishing. This phenomenon is very likely to occur when the brine cooled by such a machine is circulated in pipes through a chamber containing atmospheric air, more or less saturated with aqueous vapour, and as would actually be the case with a chamber containing fresh meat being cooled. The brine pipes under such circumstances become externally cooled with a non-conducting covering of ice, having the appearance of enamel, which, unless removed, accumulates to the extent of several inches in thickness, thus interfering with the transfer of heat, and practically preventing the room from being reduced to a lower temperature than the freezing point of water, or the melting point of the exterior surface of the crust which surrounds the pipes, whilst the brine is liable to be returned back to the evaporating ether at much lower temperatures than it should for the economical working of the machine. The same remarks apply to the employment of sulphurous anhydride and of ammonia, the limiting action in the case of ammonia, which is considered the most effective in practice, being about 35° below zero of Fahrenheit when it is employed at atmospheric tension, as in Carré's process, or in Reece's process, though of course much lower when evaporating into a vacuum, as in Professor Lindé's machine.

Almost all the statements as to the performances of these machines refer to their employment under favourable conditions, namely, the cooling of water or other fluids, or the making of ice, in which the temperature of the saline solution, or glycerine transferring the heat, never need sink below  $20^{\circ}$  Fahrenheit. When they come to be employed for cooling solids, such as masses of meat, weighing two or three cwt., to temperatures below freezing point, great practical difficulties occur in the transfer of the heat through the non-conducting air in which the meat is suspended to the pipes containing the brine, unless such pipes or other equivalent circulating apparatus are brought into close proximity to the solid masses. On board ship such arrangements are almost impossible if the ship's hold has to be employed for general cargo on the outward voyage, and in any case networks of such circulating apparatus are inconvenient and liable to leakage and to injure the cargo.

From these considerations, even if the use of dangerous chemicals on board steamers were allowable, it is apparent that cold-air machines, in which air is first compressed and then expanded, are the most convenient form of refrigerating apparatus for use at sea; and it is also evident that they are the most convenient form of machine for cooling the air of apartments generally. The great enemy of cold-air machines is friction encountered in the working of the machinery, particularly that which results in the development of heat in the expansion cylinder itself. This difficulty with friction is common, however, to all machines worked by a steam engine; viz., the Ether Machine, Pictet's Sulphurous Acid Machine, and the Ammonia Compression Machine, and the force of this remark will be understood when it is pointed out how little of the theoretical energy of a steam engine is available in practice.

It is consequently found, and generally admitted, that when an Ammonia Absorption Machine is fresh started, it gives more economical results than any other machine when the work to do is cooling liquids or making ice, and in consequence of this fact ever since its introduction by Carré about twenty years ago, it has been tried by our leading brewers and others; but various difficulties have cropped up of a very serious nature in regard to its management—the most important being the fact that when the aqueous solution of ammonia is being heated for the purpose of expelling the gas from the water, more or less steam is carried forward through the system of coils and accumulates in the vessel which should contain nothing else but anhydrous ammonia. Reece and other inventors remedied this defect to some extent, and this has been done by making the mixed ammonia gas and steam ascend a tower containing plates, arranged upon the principle of a Coffey's still, the water constantly trickling back. That an immense improvement has been effected is undoubted, and in consequence thereof leading brewers, such as Meux and Co., and Charrington, Head and Co., of London, and Guinness and Co., of Dublin, have erected large machines. But, on enquiry, I find there is not that enthusiastic approval of them which indicates they have been a complete success. For instance, Messrs. Guinness and Co.'s engineer informs me that when fresh started the machines came up to calculations, but that for three seasons subsequently they could not get more than fifty per cent., and Messrs. Meux and Co., although still working the machine, have actually since had erected an ammonia machine on the compression system, in which a steam engine is used; and in another part of their works they have recently adopted a cold-air machine, designed by myself. Messrs. Meux and Co. state that one difficulty about

the Ammonia Absorption Machine is the large quantity of low temperature cooling water required to condense the ammonia vapours, partly from the steam accidentally produced along with it. In any case the ammonia gas requires to attain a considerable pressure before it liquefies in the coils surrounded with water; if the temperature is  $60^{\circ}$ , the liquefaction takes place at 120lb. per square inch, but at  $104^{\circ}$  the liquefaction requires a pressure of 217lb. per square inch. If the boilers containing the ammonia are in a warm climate, such as that of the tropics, the liquefaction is difficult; and a case was reported recently of the blowing up of such an apparatus, twenty-eight horses being suffocated by the fumes. These difficulties have induced New York brewers to try the compression system with ammonia, the liquefied ammonia being expanded in a network of pipes, not in the vicinity of the compressing pumps, but arranged on the ceilings of the store rooms, or within their vats, the expanded gas being carried back to the compression pumps. This is reverting to a steam engine as the motive power, and brings the ammonia process into the same category as the ether and sulphurous anhydride processes. In regard to the sulphurous anhydride process, backed up by the great name of Raoul Pictet, it has had some degree of popularity abroad, but has not made much progress in this country; it is a more troublesome substance to work with than ether, and under certain circumstances, leakage and contact with moisture is actually dangerous to the metallic surfaces of the machinery, some accidents having arisen from this cause.

The ether machine was introduced twenty-five years ago, and has had a long run, but although ether is not so difficult to prepare for using in the machines as liquid sulphurous acid, or anhydrous ammonia, it is not convenient for use in hot climates, on account of the lowness of its boiling point, as compared with the heat of tropical countries.

Theoretically, according to the laws of thermodynamics, it is immaterial which chemical be employed, but the bulk of the machines, and the nature and cost of their construction, differ according to the substance selected.

The advocates of these various chemical machines have been so decided in their statements, that for purposes outside the cooling of rooms, or the carrying on of the frozen meat trade, I have not until recently thought of recommending compressed air machinery for general purposes, such as that of breweries. The well-known firm of Wm. Younger and Co., brewers of Edinburgh, have, however, recently made some experiments in this direction, and under my guidance have fitted up a Bell-Coleman Machine for cooling cellars, in which they brew and store lager beer. This kind of beer is fermented at a temperature of  $40^{\circ}$  or thereabouts, and the apartment in which the process is carried on measures  $136 \times 38$  feet in area. The machine delivers about 30,000 cubic feet of air, cooled to  $80^{\circ}$  below zero, by wooden shoots to all parts of the room, so as to maintain an even temperature of about  $40^{\circ}$ , and has been exceedingly successful for the purpose.

Although half a century ago Sir John Herschel and others directed attention to the desirability of utilizing the expansion of compressed air for the production of cold, the credit of actually constructing such apparatus appears to belong in this country to Professor Piazzi Smyth, and in America to Dr. Gorrie. As early as 1839, Piazzi Smyth had commenced small experiments, and afterwards visited a large ironworks near Edinburgh, and placed a thermometer in the diverging cone of air escaping



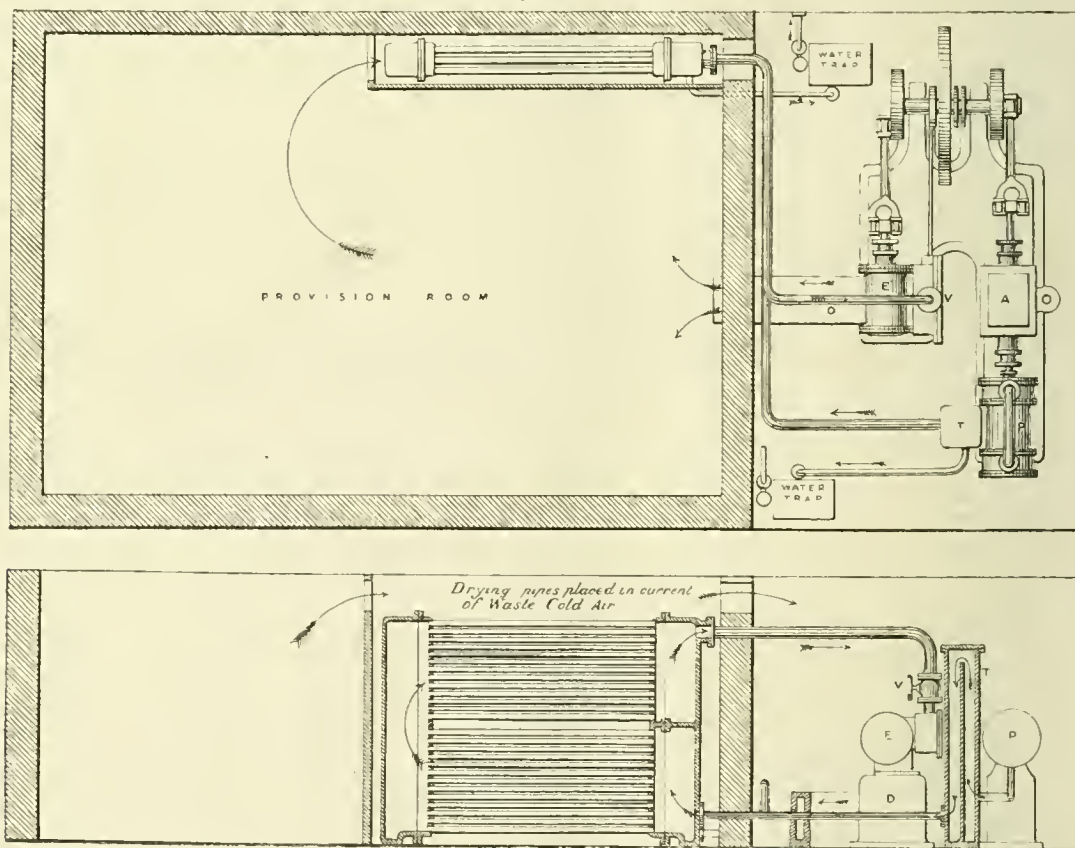
from an orifice, 1 inch in diameter, made in a large reservoir containing air compressed to the extent of about one quarter of an atmosphere, and found the escaping air to be  $29^{\circ}$  Fahrenheit colder than it was before expansion. Professor Smyth seems to have worked for a long time with apparatus on the method of blowing air through loaded valves, before the principles of the mechanical theory of heat as applied to gases were properly understood. The experimental demonstration of this theory was made by Dr. Joule in 1845, in two sets of classical experiments, and subsequently by Dr. Joule and Sir W. Thomson, in a third series of such experiments.

In the first series of experiments, Dr. Joule proved that the mean temperature of compressed air of a density of twenty-two atmospheres was not altered by being expanded into an empty reservoir of a similar size to that containing the compressed air—that is, no change of temperature occurs by the mere expansion of a gas without performance of external work, such as can be developed by letting the gas expand behind a piston. In the second series of experiments, a metallic reservoir full of compressed air was allowed to expand into the atmosphere, and it was found that the reservoir became cold in exact proportion to the force exerted by the escaping air in

From this it follows that no satisfactory apparatus can be devised by simply allowing compressed air to expand behind a loaded valve—a fact which the author has recently demonstrated.

In Fig. 1 is represented a ground plan of the refrigerating machinery erected by the Bell-Coleman Mechanical Refrigerating Company, on board the Cunard ss. "Servia." The air is compressed in the air-pump P by the steam-cylinder A, and the compressed air, cooled to the temperature of the water available for cooling, in fact, a little below this, by means of the range of pipes placed in the cold room, and is then brought to the expansion-cylinder E, where it is expanded in the act of doing work transmitted to the same shaft to which the pump and steam-piston rod are attached. Under such circumstances, the air, when discharged into the blast-box D, is about  $100^{\circ}$  colder than immediately before expansion, when the amount of compression of the air is about 30lb. to the square inch above the atmosphere. The experiment was made in this way: instead of allowing the air as usual to expand behind the working piston E, the piston-block was taken off, so that the expanding air could blow through the cylinder, without doing work, into the blast-box D, and it was found that when the air-pressure was regulated by a

Fig. 1



displacing the atmosphere. In the third series of experiments made by Dr. Joule and Sir W. Thomson conjointly, it was proved that when compressed air was expanded through small orifices into the atmosphere, the cold developed by the force exerted in displacing the atmosphere was almost entirely counterbalanced by the various frictions encountered by the molecules of air before coming to rest after expansion.

throttle-valve V, no lowering of the temperature of the air in the blast-box could be detected, when at exactly similar air-pressures, but with the expansion taking place in the act of doing work propelling the piston,  $60^{\circ}$  reduction of temperature occurred. The air-pressure at which the experiment could be made was less than the normal working pressure of the machine, owing to the crank-shaft being deprived of the assistance of the expansion cylinder.

Professor Rankine, in a paper communicated to the British Association in 1852, calculated that 25,000 cubic feet of air should be cooled per hour from 90° to 60° by an engine of 1 HP., allowing for friction as in a Cornish engine; but he advocated avoiding friction as much as possible, by effecting the compression of the air by a bell-shaped gas-holder being depressed into a reservoir of water, and expanding it back again to its initial pressure in a similar shaped vessel attached to the opposite end of an oscillating beam, by which means, he said, 66,000 cubic feet should be reduced 30° Fahrenheit by 1 HP. per hour. In the same year, Sir William Thomson went independently into the subject, and specified the size of compression cylinder and of expansion cylinder, which are theoretically required to cool 1 lb. of air per second (15.5 cubic feet), from 80° to 50° Fahrenheit, and also the power required to do this amount of work, which he specified to be 0.288 HP., supposing there to be no loss of effect from friction. This, it will be observed, is equivalent to 193,750 cubic feet per hour, reduced 30° Fahrenheit by 1 HP. per hour. Sir William Thomson also pointed out in the same paper an arrangement which would be equally suitable for heating buildings, proposing that air should first be rarefied so as to be cooled a little, and then passed through tubes surrounded with a current of cold water (say from the town supply), and from which the slightly rarefied air could pick up heat, and being restored to atmospheric pressure, would be to a corresponding extent warmed. By this process it is possible to warm air through a range of 30° with one-third of the coal which would be required by using it directly, if all the heat escaping in any way from the engine or from the fire be added to that which is picked up by the tubes surrounded by water. Sir William Thomson has also remarked recently that this may be the future of heating buildings, when such sources of energy are available as that of the Falls of Niagara, for working the machines, and is quite as likely to be satisfactorily solved as has been the reverse operation of cooling.

A practical and theoretical discussion of the subject was undertaken by Professors Rankine and Piazzi Smyth jointly, and lasted from 1851 to 1856, being chiefly with reference to the cooling of buildings in India, and for a particular military hospital—the idea of Professor Piazzi Smyth being to employ hand or animal power for the work. The investigation effectually demonstrated that the cold produced by the compression and subsequent expansion of air, unless carried to higher pressure than can be attained by the mere depression of a bell-gasometer into a tank of water, is useless for practical purposes, as in order to get a compression of one atmosphere, such tank would have to be 34 feet high. It was found that though this suggestion of reducing friction, which is the great enemy of cold-air machines, to a minimum was effectual, there was another enemy, viz., the influence of the heat evolved by the condensation of the aqueous vapour, owing to the air being reduced in temperature. Thus 20,000 cubic feet of saturated air at 90° Fahrenheit contains 47 lb. of water, but only 17 lb. at 60°, so that the latent heat liberated by the condensation of the water, even although the air is not perfectly humid, entirely neutralises the effect of the compression to a fraction of the atmosphere.

The question of cooling buildings and hospitals in India remains much as it was left in 1856, but as more experience has now been gained of a practical character in the working of such machinery, it is to be hoped that its importance to the health and comfort of the Queen's subjects in India, and other tropical countries, will not be overlooked.

Dr. Gorrie's machine was not a cold-air machine in the sense that the word is now used—that is, a machine for cooling air and then delivering it into apartments for the purpose of ventilating and cooling them—it was a machine for cooling brine or some other liquid which would not freeze easily, and which in its turn could be used in making ice. According to information supplied to me by Mr. James Brownlee, late of New Orleans, and now of Glasgow, the first machine was constructed in New Orleans before the year 1845, and consisted of a cylinder about eight or nine inches in diameter for compressing air, and of another cylinder about two-thirds the capacity of the first, in which the air was expanded. A jet of water was injected into the compressing cylinder, and the compressed air was discharged into a vessel surrounded by cold water, where it was further cooled, and the injected water allowed to settle. The air was then admitted to the expansion cylinder, and whilst expanding a jet of salt water was injected. The salt water being drawn off, was allowed to circulate around vessels containing fresh water to be frozen. In the year 1850, a patent was taken out in Great Britain by Mr. Newton, the patent agent (communicated from America), the process being identical with the one just described. Dr. Gorrie died shortly after the appearance of this patent, which does not seem to have been reduced into successful practice.

In the year 1857, Dr. C. W. Siemens obtained provisional protection for an invention which was not completed; it consisted in combining with an arrangement somewhat similar to that of Dr. Gorrie's, an "interchanger" for the purpose of subjecting the compressed air before expansion to the waste cold air travelling back to the pump to be re-compressed with a view to economise cold, but no method was specified for removing the moisture which such an arrangement would cause to be deposited from the condensation of the aqueous vapour in the air, nor does it appear that the patent was ever completed or put into practice.

In 1862 appeared Mr. Kirk's machine, and which was also a machine designed for cooling liquids or making ice; and the details of which, as also those of the remarkable preliminary experiments, in which mercury was frozen, are as interesting as the practical success of his machine was marked.

In 1869 two German engineers, Messrs. Windhausen, of Brunswick, and Nehrlich, of Frankfort-on-the-Main, took up the matter of cold-air machines with great vigour, re-patenting a great deal that was old in almost every country in Europe, and they were followed by Mr. Paul Giffard, in 1873. Windhausen's patent of 1869 directed the compressed air to be cooled in pipes surrounded by cold water. Giffard's of 1873 directs it to be cooled by injecting water into the pump. Windhausen having tried surface cooling, reverted to water injection into the pump in a patent of 1873. Nehrlich, in 1874, went back to surface cooling. Giffard adopted direct injection in 1875, and then returned to surface cooling in 1877. The fact is that, whichever method was adopted, the discharged air was always loaded with snow to an inconvenient extent.

The reports\* of Dr. A. W. Hoffman in connection with the Vienna Exhibition of 1873, together with the documents submitted to the jurors of that exhibition, stated that a large Windhausen machine, capable of discharging 150,000 cubic feet of air per hour, was being made for New Orleans, and one of about 80,000 cubic feet per hour for the brewery of Mr.

\* *Vide* Berichte über die Entwicklung der chemischen Industrie während des letzten Jahrzehnts.



Peter Overbeck, of Dortmund, and one of 80,000 cubic feet per hour for the brewery of Messrs. Hildebrand, of Pfungstadt.

In regard to the first-mentioned machine, Dr. Hoffman's report states that it was tried in Berlin before being sent out to New Orleans, and although the compressed atmospheric air was cooled in pipes surrounded by cold water, and not by direct injection of water, it was found that when the air passed into the expansion cylinder, the greater part of its existing vapour was deposited as snow, which interfered with the working of the machine, and choked up the escape pipes. On arrival in New Orleans it worked badly, and was dismantled. This was also the fate of the machine put up at Overbeck's brewery; but that at Hildebrand's brewery is still in occasional use, being worked off an engine used for other purposes. A few other Windhausen machines are in existence; for instance, one in Fairbank's lard refinery, in Chicago, which, I have been informed, has never been entirely satisfactory, and has been frequently altered. It can only be worked at low pressures, and at present it yields air at about 14° Fahrenheit, and has a compressor of 42 inches diameter and 36 inches length of stroke, and an expansion cylinder of 36 inches diameter and 36 inches length of stroke.

Latterly Windhausen has turned his attention to refrigeration by the rarefaction of air in contact with sulphuric acid.

Matters were in this position in the beginning of 1877, when my attention was directed to the subject by Sir W. Thomson, who had been consulted by Messrs. Henry and James Bell, of Glasgow, as to the possibility of constructing machinery to supersede the use of ice in the preservation of fresh meat during its passage across the Atlantic.

A small experimental machine of the Giffard type, since discarded, and resembling in appearance a little vertical donkey pump, was in existence at the works of Messrs. Hick, Hargreaves and Co., Bolton; but a review of the hundred or so previous patents on the subject, of the practical failures of the German engineers, and of the fact that no cold-air machines had got into regular use on land, not to speak of making one suitable for ships' use, was anything but encouraging.

An attentive consideration of the matter upon general principles led to the following conclusions:—

1. That atmospheric air is really not air alone, but a mixture of aqueous vapour and air, and that when such mixture is compressed into pipes surrounded externally by water of the same temperature as the air before compression, the invisible vapour of the air becomes condensed in the direct ratio of the compression, in virtue of the law of physics demonstrated by Dalton, and expressed by the statement that a cubic foot of air in contact with water contains exactly the same weight of vapour, whatever may be the density of the air, temperature being kept constant. If the density is increased, the vapour liquefies—if it is diminished, water evaporates into the air.

2. Compressed atmospheric air of usual humidity is not therefore made wetter by injection of water, provided the surplus water is run off continuously by automatic traps, air being actually dried by compressing it in contact with water, removing the water and expanding it.

3. Direct injection is the quickest and most effective method of cooling air to the temperature of the water, which is a condition necessary to the working of a machine with the least expenditure of power.

4. Injection of a shower of water into freshly compressed air tends to settle the fog caused by the sudden condensation of the invisible atmospheric vapour, thus facilitating its removal by traps.

5. That whilst the direct injection of water is desirable for cooling the air to the temperature of the water, it is not absolutely essential, if the compressed air be passed through a sufficient number of pipes surrounded by cooling water, the ultimate result in either case being that the compressed air can only be reduced to the temperature of the water, which is not sufficient to liquefy the vapour usually contained in the air, except the air pressures employed be excessively high—which is fatal, according to the first principles of thermodynamics, for working a machine economically.

6. That every lb. of vapour unnecessarily condensed liberates as much heat as will raise about four thousand times its weight of air 1° Fahrenheit, and that air absolutely dry is a condition that would abstract the fluids of animal tissue, and indeed is a phenomenon unknown in nature, the degree of humidity being generally over 50°, even in what is, in common parlance, called "dry air."

7. That a convenient way of liquefying such vapour is to apply a portion of the cold air produced by the machine to the external surface of the pipes or other vessels conveying the compressed air already cooled by water to the cylinder in which it is to be expanded the liquid condensed in this way being removed by automatic traps.

The last principle does not seem to have been applied in practice in this country prior to my adopting it in conjunction with Messrs. Bell. It is true that an "interchanger" was suggested by Dr. Siemens in his incomplete patent of 1857, in Messrs. Laidlaw and Robertson's patent of 1864, and in Mr. Windhausen's of 1869, for intensifying the cooling power of the machinery, by the utilisation of the waste cold, after air has been used for freezing water; but in reality there is no waste cold air if rooms of graduated temperature are being cooled; neither would a simple heat exchanger prevent the formation of snow or ice in the expansion cylinder unless the vapour condensed by such interchanger were removed with regularity, for which no provision was made.

In attempting to carry these principles into practice, I was met with another set of conditions from the practical engineer who had to use the machines, and particularly the marine engineer—such as the necessity of having everything strong, with plenty of rubbing surfaces and material to stand wear, and the avoidance of using parts requiring frequent replacements, such as light piston rings, all of which, whilst favourable for a machine being worked continuously night and day for three or four months, are quite inimical to the reduction of friction.

The first machine constructed by the Bell-Coleman Mechanical Refrigeration Company of Glasgow, under the guidance of myself, was built in 1877. It was intended entirely for marine work, and to work between the decks of a vessel where the vertical height does not exceed 6 feet 6 inches; it was also to be duplicated in all its parts, in fact, a double engine on one sole plate, so that if one half broke down the other could be worked by itself. This machine consisted of two steam cylinders, 10 inches in diameter and 18 inches length of stroke, two air-expansion cylinders, 14 inches diameter and 18 inches length of stroke, and four air pumps, of 14 inches diameter and 18 inches length of stroke, all connected by a four-throw crank-shaft, with four piston-rods.

An amount of water equivalent to five times the weight of air taken in was partly injected by the water-pump into the compressors, and partly in the form of a shower of rain into the air immediately after leaving the compressors, by which it was reduced to a temperature within 2° or 3° of that of the water, the latter being drawn off by automatic traps or ball-

cocks. The air was then subjected to some of the cold air produced by the machine itself, by being made to traverse copper pipes arranged around the interior of the walls of the room of about 20,000 cubic feet capacity, being cooled. It was found that about 588 square feet of surface in the pipes caused a lowering of about 20° in 20,000 cubic feet of air passed per hour. The moisture liquefied by this arrangement was removed by automatic traps, and was sufficient to prevent any inconvenient amount of snow being formed in the expanded air when the working pressure of the air was 30 lb. per square inch above the atmosphere, the air being previously cooled by water to 65°. After this the air was expanded and delivered by means of wooden shoots to the various parts of the meat room. The temperature of the meat room was 35°, the external air being about 70° Fahrenheit. A wet-and-dry-bulb hygrometer suspended in the room never indicated more than 70 per cent. humidity during the continuous working of this machine for two months. Several carcases of beef were preserved in splendid condition, and fish became stiff and their wet skins externally quite dry by a day or two's exposure to the atmosphere of the room. It was found, however, that the ratio of the steam cylinder capacity to that of the compressors was not such as to allow of the machine being worked with steam of 40 lb. without the assistance of a steam-condenser, a condition which was essential on shipboard, as the full boiler pressure and the use of the condenser of the main engines could only be obtained at sea, and the machine was required to work in port occasionally.

Neither was the design so compact as was thought desirable, and it was then decided before putting anything on board ship to adopt a design which was put on board the Anchor Line steamer "Circassia," in March, 1879, and connected with a chamber measuring about 18,000 cubic feet, including engine space and chamber walls. The moisture-depositing pipes of copper, which were to be exposed to the cold air of the chamber containing the meat, were arranged exactly fore and aft, so as to prevent their action being interfered with by the roll of the ship, the pitching of the ship being a lesser evil.

The author went to New York with this machine to watch its behaviour, and returned in the following month with a small cargo; the machine was able to keep the room near freezing-point with sixty revolutions per minute. He returned to New York and back again with meat to the extent of four hundred carcases of beef and large quantities of mutton, equivalent in value to about £8,000 sterling. This meat had been previously cooled to 35° in the chill-rooms of New York, and was landed here at about the same temperature. The machine ran a great number of voyages with similar cargoes, and finally giving place to a larger machine, was transferred to another steamer.

It may, however, be mentioned that in July, when the temperature of the harbour-water in New York was 85°, and the external air over 100°, it took all the power of the machine working at one hundred revolutions per minute to do the beforementioned work, which has been the basis of all subsequent calculations for a given room with such insulation as was then used. Matters had now arrived at the stage when it was deemed desirable by the owners of the Anchor Line to fit up the whole of their thirteen Transatlantic ships trading between this country and New York. After much consideration it was resolved to abandon the construction of machines in duplicate, and to trust to making a machine with an unusual factor of strength together with abundance of spare gear.

The design fixed upon is shewn in Fig. 2. The

first machine, however, of this type was placed on board the steamship "Strathleven," chartered by Messrs. McLlwraith, MacEacharn and Co., for an experimental voyage to Australia. The two compressors marked P P were 16 inches in diameter and 24 inches length of stroke, the steam cylinder A was 18 inches in diameter and 24 inches length of stroke, and the expansion cylinder E 16 inches in diameter and 24 inches length of stroke. The moisture-depositing pipes, for conveying the compressed air from the compressors to the expansion cylinder with these machines, were made of galvanized wrought iron expanded into cast-iron tube-plates, the top rows of tubes bringing back the compressed air from the far end of the pipes placed in the cold-room. These pipes were generally ranged fore and aft in the centre line of the ship, but in some cases, as in those of the "Strathleven" and the "Cuzco," they have been arranged next the ship's skin; they do not occupy, as a rule, more space than 300 cubic feet in a chamber of the gross measurement of 20,000 cubic feet, or a floor space of about 1½ per cent. to 2 per cent. of the total area of the room. The machine-room seldom exceeds 7½ per cent. of the space being cooled, including working space round the machine. In summer these machines have been able to reduce in a few hours in New York Harbour, with air and water temperatures of nearly 100°, the air of chambers 60 feet long, 40 feet broad, and 6 feet high, to below freezing-point, and at other times of the year they have been employed with equal success for chambers 100 feet long, 40 feet wide, and 6 feet high, internal measurement. Their average speed during a voyage has been about fifty to sixty revolutions per minute, and their average working air pressure 45 lb. per square inch above the atmosphere.

As regards the one fitted on board the steamship "Strathleven," it arrived in London in February, 1880, bringing 34 tons of mutton and beef frozen solid, being the first successful importation of fresh meat from Australia. The machine was only working a fractional portion of each day during the voyage, being connected with a chamber measuring internally about 4000 cubic feet. Subsequent experience demonstrated that the same machine could have cooled a chamber of four times the size. In the case of the "Strathleven" and other Australian ships, the wall insulation was increased to 10 inches, and was formed of hollow walls filled with wood charcoal.

The log of this machine was kept by Mr. Matthew Taylor Brown, B.Sc., Glasgow, who went out to Australia in charge of the machinery. It contains much interesting information, particularly observations upon the minimum temperature of the air immediately after expansion, which fell to 100° below zero, with an air-pressure of four atmospheres absolute, and cooling water of 50°. Mr. Brown also made observations upon the freezing operations in Australia, and found that when a quarter of beef at a temperature of 70° was put into an atmosphere of about 10° Fahrenheit, its temperature at the expiry of five hours was 39° at the surface and 55° in the centre, and that twenty-four hours elapsed before the surface was frozen hard, the portions 6 inches below the surface being only about 50°; in four days, however, the centre of the mass became reduced to a degree or two below freezing-point. It was also found that the conditions of bringing meat from Australia are very favourable for the working of the machinery, since the long run from Melbourne to the Equator in temperate waters allows the machine to get a thorough mastery of the cargo before arriving at the regions of the Red Sea.

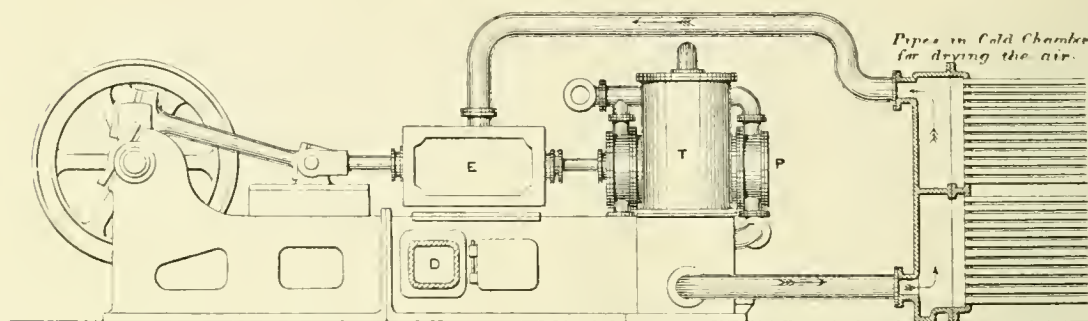
Steamers and sailing vessels fitted up with machinery on the lines laid down in 1878 are now



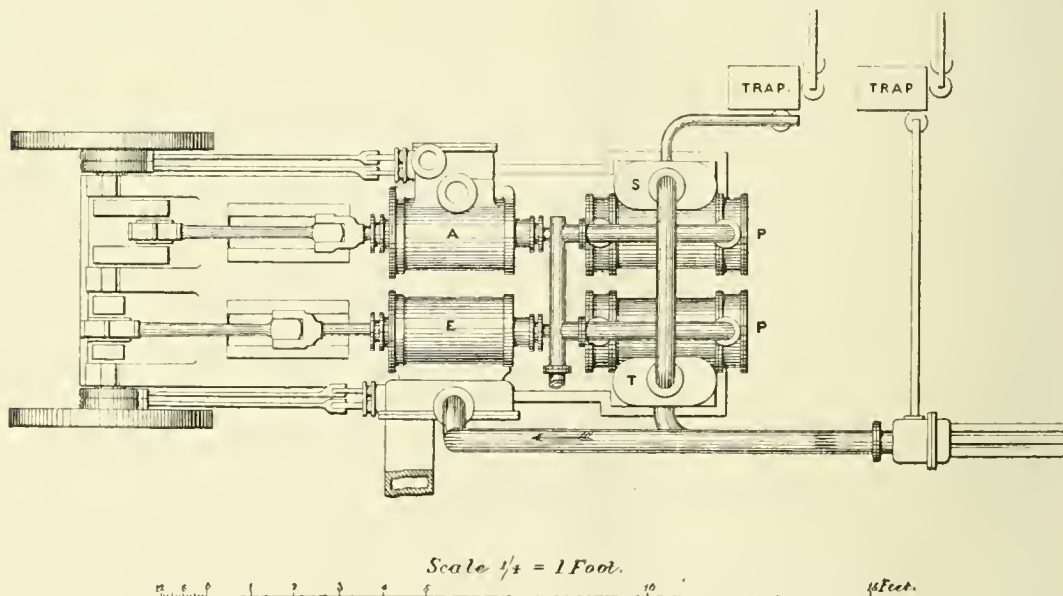
to be found regularly traversing the Atlantic and Pacific, and frequenting the ports of London, Glasgow, Melbourne, Sydney, Canterbury, Otago, Batavia, Calcutta, New York, Bermuda, and crossing the Red Sea, the Indian Ocean, and the Mediterranean. Their aggregate cooling power is such that they could

ments; they have again transferred it to the shores of Australia, where it has finally appeared perfectly fresh and in splendid condition on the tables of the merchant princes of Melbourne and Sydney. They have conveyed a great number of tons of English fish by means of the Orient steamers to the same colonies,

Fig 2



NOTE.—In Figures 2, 4, 6, and 11 the position of the moisture depositing pipes and the lead of the other pipes are arranged so that the currents of air may be clearly shown and slightly different from the arrangements actually adopted in practice which vary with each ship.



without difficulty freeze 400,000 tons per annum, and, if worked at full power and speed, their steam cylinders would indicate full 8000 horse-power. Their work is various. They have brought frozen salmon from the shores of Labrador and delivered it in London; they have kept it in the frozen state in London for six months whilst being sold in instal-

comprising soles, turbot, whiting, etc., which have been bought up and eaten by our Australian cousins, curious to taste and eager to pay several shillings a pound for such dainties. Their chief duty has, however, been in bringing meat to Great Britain, upwards of 400 cargoes having arrived at our ports of the money value of over three millions sterling.

Fig. 3 represents machines as now constructed for cooling ships' provisions and making ice. The air is compressed in the air-pump, P, deprived of its heat by compression in the tower, T, passes through the horizontal moisture-depositing pipes packed in the sole-plate, and is finally expanded in the act of doing work in the cylinder, E, from whence it is conveyed round the outside of the metallic cells containing the water required to be frozen for table use, and then is delivered into the chamber containing the meat and provisions. The length of this machine is 12 feet, its breadth at the point P is 3 feet, and at the point E is 4 feet, and its height is such that it will go in 'tween

they assume when packed in ice. The provision-room of a large steamship measures internally perhaps 2000 cubic feet, including the space necessary for storing the wines, salt meat tanks, etc. This pattern of machine is proving a very useful one in another direction, for, by arrangement with Messrs. Crossley Brothers, the makers of the Otto gas engines, they are being constructed to be driven by gas instead of by steam. The air-pump in this new machine is placed in the position of steam cylinder, Fig. 3, and the gas motor cylinder takes the place of the air-pump, P, on same diagram. Such machines, affording a cooling power equal to what could be obtained by

Fig. 3.

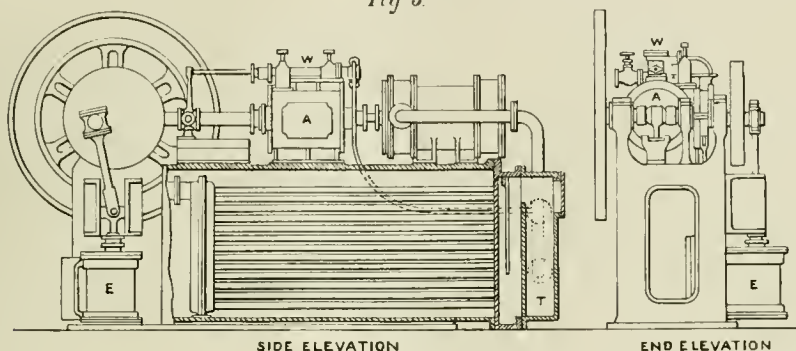


Fig. 3a.

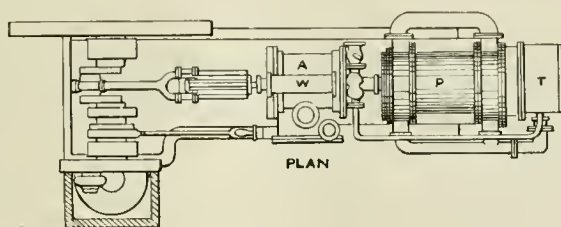
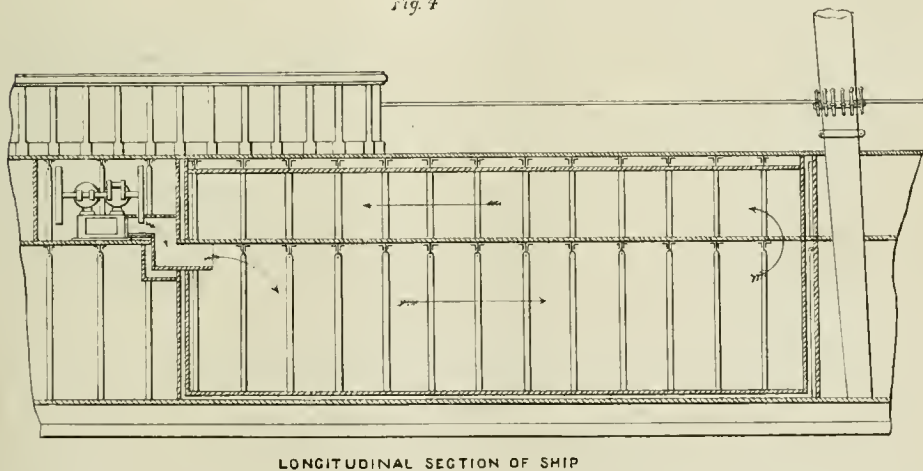


Fig. 4



decks of any vessel. Its best position is adjoining the main engine-room, but as the stewards' departments are frequently elsewhere, the machine is sometimes placed on the upper deck, or in the position usually occupied by the old ice-house. The air delivered by these machines is so dry, as well as cold, that calico or paper hung up in it ignites readily on application of a flame, so that the provisions are prevented from getting into the slimy condition which

the use of half a ton or a ton of ice per day, are now being made for retail shops, country mansions, etc.

On diagram 4 is a section of one of Messrs. Shaw, Savill and Albion Company's ships fitted up, the end view of the engine appearing in top left-hand corner, and the space cooled being not only the 'tween decks, but the lower hold of the forward half of the vessel.

In the case of the steamships of the Orient Line and other steamers, the cold chambers for containing



the meat have been constructed in the 'tween decks of the ship, so that their form varies; but the method of building them consists of lining the walls, the floor, and the roof of the meat-chamber with wood-work containing hollow spaces filled with wood-charcoal, or shavings. It is found that 7 inches thick of such lining forms very fair insulation; but, in the case of tropical ships, it is frequently made 10 or 12 inches thick. It is also found that the larger the quantity of meat brought over, the more economically can the work be done. Thus the prime cost of the machinery for carrying a pound of meat is 25 per cent. less when a ship is fitted up to carry 1000 tons instead of 500 tons, and there is a further gain of 10 per cent. in the less packing space required.

The air discharged from the machines is generally 80° below zero Fahr. and oftentimes 100°.

The lowest temperatures which have been obtained in scientific laboratories has been about 200° below zero, but it is believed that before 460° below zero is reached, all known gaseous matters would be absolutely solid.

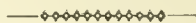
The fact of air being so excessively low in temperature when first liberated from the machine does not in any way interfere with keeping a regular temperature in the cold chambers—in fact a temperature so regular that nothing even in the most equable climate can possibly be compared to it. An examination of thousands of recorded observations of the machines traversing the Atlantic shows that the temperature of the chambers does not vary 3° during a voyage, being much less variation than the difference between the hours of morn and even of any ordinary winter day.

This even temperature is maintained by distributing the cold air by pipes carried along the top corners or roof of the chamber, and having outlets at regular intervals, the velocity or flow of the air being regulated by the speed at which the machine is worked. There is no big blast or current of air perceptible at these outlets, the cold air tranquilly displacing the warmer atmosphere as easily as cold winter air permeates through the chinks of windows and doors of a sitting or drawing room, and the atmosphere is no more disturbed than in such cases. It follows from this that a chamber can be kept at any temperature deemed desirable—thus 20° Fahr. is thought a very desirable temperature for bringing meat long distances across the tropics, 28° Fahr. is thought desirable for bringing meat from America, and 40° Fahr. for preserving town meat in public abattoirs; whereas in India the temperature required for sitting rooms is perhaps 70° or 80° Fahr. There is no difficulty in meeting any of these conditions; and, in regard to India, I will quote from the Calcutta paper "The Englishman," of Feb. 2nd, 1884, regarding a refrigerator and cold sitting-room exhibited at the recent Calcutta exhibition. It says—"This is one, and perhaps its strongest, point of usefulness from a commercial point of view, but it has another career from a scientific and humanitarian point of view, and that is in connection with the surgical department in hospitals. In tropical climates there are many surgical operations which cannot be performed until the weather gets cooler, a certain temperature being a *sine quâ non* to the success of the operation, so the unfortunate patient has to remain in suffering, whilst the disease is daily gaining ground, because the weather is too hot to admit of the operation being performed; but with a Bell-Coleman cold house the surgeon can command any temperature he wants for the operation, and maintain it as long as may be necessary for the recovery of the patient. Dr. Tuson, the Deputy Surgeon-General, has made frequent visits to this one, becoming each time more convinced of its adaptability to hospital purposes, and to the enormous

advantages such a room would confer in certain operations. It seems to be becoming a popular place of resort, as upwards of 2000 people have been inside already, and appear surprised to find Darjeeling or Simla so easily attainable."

Whilst it happens that on the Continent of Europe other kinds of refrigerating machinery have been extensively employed, the exigencies of the meat trade have so far developed cold air machines that they are more extensively used in this country than any other kind of refrigerating machinery. The readiness with which consignees of meat trust 10 or 20 thousand pounds' worth of that commodity to the working of a single machine, out in the ocean for two or three months is a remarkable fact, and speaks strongly in favour of their reliability. Moreover the absence of chemicals other than air and water, renders them easily repaired by a mechanic who understands a steam engine, of which such may be found in almost any foreign port.

Their use, however, has not been confined to sea-going purposes, but has embraced a number of applications on land, besides such obvious purposes as the cooling of slaughter-houses, and the cellars of brewers, amongst which may be mentioned the keeping of rooms cool in which oil is being filtered or pressed—and no doubt numerous possible applications will suggest themselves to the members of this Society.



## ON THE QUANTITATIVE ESTIMATION OF OILS AND FATS (PART II).

BY EDMUND J. MILLS, D.S.C., F.R.S., AND THOMAS AKITT.

In a preceding memoir (Mills and Snodgrass, *Journal* ii., 435), a method was described of quantitatively estimating oils and fats by dissolving these in carbonic disulphide, and titrating with a solution of bromine in the same solvent. Further experience with this method has convinced us of its practical value in such estimations; we have therefore sought to improve it, and to extend the range of standard bromine absorptions. The principle of the method consists in using a common solvent (non-aqueous) for the bromine and the oil or fat; and we continue to lay special stress on the exclusion, as far as possible, of water during the absorption of the bromine.

As regards the solvent itself, we now employ carbonic tetrachloride instead of the disulphide. The former liquid has of course an advantage over the latter in point of odour; a more material superiority lies in the fact that a solution of bromine in the tetrachloride has much greater stability at the ordinary temperature than is the case with the disulphide.

The boiling points of the disulphide, bromine, and the tetrachloride are respectively about 43°, 63°, and 77°C.; and a mixture of the three bodies might possibly be so made as to contain a percentage of bromine perfectly constant at the ordinary temperature. The tetrachloride solution, however (with which we now always work), has at least twelve weeks' exact stability; sufficient, at any rate, for all practical requirements.

Nearly all oils and fats, when tested in the natural state, contain moisture which is difficult to remove completely. When the disulphide is the solvent, the influence of this, even if 0.5 gramme of the substance be taken, seldom produces an increased absorption of bromine; but the tetrachloride, as might have been expected, does not so effectually exclude the oxidising effect of moisture, and not unfrequently requires the amount of oil or fat to be restricted to 0.1 gramme or less.

The fact that oxidation of this kind is occurring is easily ascertained by the requirement of an increased percentage of bromine, as the weight of substance is increased.

Dissolving, then, about this quantity of the sample in 50 c.c. of tetrachloride, we add standard bromine until there is, at the end of fifteen minutes, a permanent colouration. This we compare with a colouration, similarly produced, in a blank experiment, and so obtain a measure of the bromine absorption; or as we sometimes prefer, titrate in the ordinary manner by adding the solution of the brominated substance to potassic iodide with starch, and sodic thiosulphate to this, or the converse.

It naturally occurred to us to endeavour to find a substance by which the excess of bromine might be "titrated back" in the same solvent, and thus reduce the whole of the work to the same degree of simplicity as a common volumetric estimation of alkali. After trying fruitlessly a large number of bodies, we at length, towards the close of our work, found in  $\beta$ -naphthol the substance of which we were in quest. In presence of carbonic tetrachloride, we find that  $\beta$ -naphthol forms a monobromo-derivative; so that our solution of it is made up to correspond with the bromine solution in the ratio  $\text{Br}_2 : \text{C}_{10}\text{H}_8\text{O}$ . The "absorptions" stated below, of Japan wax, myrtle wax, Carnauba wax, and common resin, were determined in this way, the excess of bromine being discharged by standard  $\beta$ -naphthol. It may not be unimportant to add that  $\beta$ -naphthol is readily and cheaply procurable.

An artifice which we have sometimes employed in reading the colorimetric effect should perhaps be mentioned here. The yellow colour produced by the action of the bromine on an oil, for instance, occasionally obscures the observation of the red tint of the slight excess of reagent. When this has happened, we have taken the reading through a solution of potassic chromate interposed between parallel glass plates, or inserted in a test tube. This plan was specially had recourse to in the case of skate, ling, and shark oils.

In a forthcoming paper it is our intention to deal with the subject of resins.

We cannot conclude the present memoir without expressing our thanks to Messrs. Field, Henderson, Parker, Whitelaw, W. B. Dick and Co., and others for specimens; and particularly to Mr. Hay Henry for a beautiful collection of fish oils. We are also much indebted to Mr. J. Muter, who assisted us in connection with the determination of the specific gravity of the oils and the melting points of the waxes, &c.

A table of results is subjoined. Each number (except in the case of Japan wax I) is a mean of three determinations.

The average probable error per cent. of a single result is 0.62; where  $\beta$ -naphthol was used as an accessory, this was reduced to 0.46. Mean strength of the standard bromine, 0.0644 grm. per c.c.

Substance.	Absorption. %	Specific Gravity at 11°-12°.	Melting point.	Remarks.
Almond Oil ....	26.27	.9168	..	Exp. from bitter almonds
" ..	53.71	.9151	..	Exp. from sweet almonds. Yellowish (1883). Very yellow
Beeswax .....	0.54	..	63.9	Scotch (1876). Pale
" ..	0.00	..	63.2	Scotch (1882). Yellow
" ..	0.00	..	62.9	Scotch (1883). Yellow
Ben Oil .....	52.95	.9198	..	Much solid fat
" ..	50.89	.9161	..	No solid fat
Carnauba Wax ..	33.50	..	81.1	1877. Scotch, rancid with age
Cod Oil .....	83.12	.9269	..	1882. Norwegian refined
" ..	81.03	.9292	..	1882. Japanese
" ..	82.91	.9257	..	1882. Scotch
" ..	81.61	.9277	..	1883. Crude, from liver refuse
" ..	86.69	.9281	..	1883. Norwegian
" ..	83.01	.9318	..	1883. Scotch
" ..	82.07	.9278	..	
Croton Oil .....	46.66	.9441	..	
Eucalyptus Oil ..	91.09	.8691	..	
Horse Fat .....	35.67	..	..	Pasty, well mixed
Japan Wax (D. (II) ..	2.33	..	50.5	
" ..	1.53	..	50.8	
Java Nut Oil ..	30.21	..	..	
Ling Liver Oil ..	82.44	.9295	..	1882
Maize Germ Oil ..	74.42	.9262	..	1880
Mustard Seed Oil .....	46.15	.9152	..	East Indian
Myrtle Wax ..	6.34	..	41.3	
Neatsfoot Oil ..	38.33	.9147	..	Thick
Niger Seed Oil ..	35.11	.9214	..	
Olive Oil .....	59.31	.9266	..	Thick, brown, "best sulphocarbon"
" ..	60.61	.9382	..	Thinner, greener, "low quality sulphocarbon"
Palm Oil .....	35.41	..	..	Crude old Calabar
" ..	34.96	..	..	Crude Lagos
Peach Kernel Oil .....	25.40	.9175	..	
Poppy Oil .....	56.51	.9214	..	Turbid, filtered
Resin (Common) ..	112.70	..	..	Light colour
Seal Oil .....	57.31	.9241	..	Pale
" ..	59.92	.9216	..	Dark
Sesame Oil .....	47.35	.9250	..	
Shark Liver Oil .....	81.36	.9293	..	Dec. 1883
Sunflower Oil ..	54.32	.9391	..	Colourless. About 1883
Whale Oil .....	30.92	.9199	..	"Norwegian white whale." Very thick
" ..	48.69	.8780	..	"Bottle nose whale"

<sup>1</sup> One hour's absorption, clear portion taken.

<sup>2</sup> Twenty hours' absorption.

<sup>3</sup> One quarter of an hour's absorption.

#### DISCUSSION.

Mr. STANFORD (chairman) said: This method of Professor Mills is a great improvement on what we have heard before, especially as I can understand the value of the absence of water in treating these oils. There are several members present who are interested in this subject, and we shall be glad to hear anything they may have to say.

Mr. A. H. ALLEN (of Sheffield) said: I have listened with great interest to what Professor Mills has said respecting his improved process for estimating oils and fats. I am not surprised to find that he has abandoned the use of carbon disulphide, for he is well aware that the objection which was raised to it was not based merely on the smell, but also on physiological grounds. In fact, when using his former process in my laboratory, very disagreeable effects were observed. I can endorse his statement as to the desirability of working in the absence of water in certain cases, and I congratulate Dr. Mills on having found a suitable indicator for the end of the reaction. This, and the replacement of the disulphide of carbon by the tetrachloride, will probably remove the prac-



tical objections which existed to his former process. The one disadvantage which occurs to me in using the tetrachloride is the employment of a substance not in everyday use in the laboratory, and I should like to learn whether Dr. Mills has examined the applicability of a bromised petroleum spirit; as, if such a solution proved to be fairly stable, it would be cheaper, and in some respects more convenient than the solution of bromine in tetrachloride carbon.

Professor MILLS, in reply, said: As regards the question of aniline and toluidine, we find that we are perfectly able to distinguish in analysis by means of the tetrachloride process; but we have not had time to complete our experiments with them. As regards the use of petroleum spirit, of course, for cheapness' sake, one should naturally prefer it, as the chloride of carbon is not a cheap substance; but if you once have a stock of it, it can be re-distilled with very great ease. The reason why we do not adopt petroleum spirit is that we believe that, in all probability, bromine would act upon petroleum spirit, even if this had been carefully freed from all unsaturated hydrocarbons. As a solvent for oils and fats petroleum would have been unexceptional; but as a means of keeping bromine solution unaltered for a long time it would probably fail. Chloride of carbon, however, is absolutely stable towards bromine at least for six weeks, and probably much longer.

## Journal and Patent Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Secret Specifics against Boiler-Incrustations.* Dingler's Polyt. Journ., 251. Heft. 12. from Ztsch.d. Verbandes der Dampfkesselüberwachungsvereine.

THE following analyses and statements are compiled from reports due to the engineer, H. Bellmer, in Stuttgart, and to the "Heizversuchstation des Bayerischen Vereines, Lapidolyl," made by Gebrüder Kolker, Breslau; four specimens from different sources, contained in one litre.

	I.	II.	III.	IV.
Total residue ..	85.67 g.	68.23 g.	92.73 g.	64.42 g.
Na <sub>2</sub> CO <sub>3</sub> ..	37.77 ..	9.64 ..	34.48 ..	11.58 ..
NaHO ..	10.60 ..	9.60 ..	11.66 ..	13.60 ..
Combined Na ..	0.85 ..	8.75 ..	— ..	— ..
Organic (Catechu) 28.81 ..	31.10 ..	35.98 ..	31.44 ..	— ..

"Anti-incrustant solution." Maker H. Patrosio in Bologna, two specimens contained per litre—

	I.	II.
Na <sub>2</sub> CO <sub>3</sub> ..	71.464 g.	84.300 g.
NaHO ..	6.080 ..	15.200 ..
NaCl ..	28.952 ..	8.120 ..
Na <sub>2</sub> SO <sub>4</sub> ..	7.830 ..	5.000 ..
Organic ..	72.084 ..	27.168 ..
	185.860 ..	139.788 ..

"Anti-incrustant powder." Maker N. Albert in Berlin; according to the following analysis, this is similar to the so-called "Paralithicon Minerale." (See 1876 [220] 265.)

CaCO <sub>3</sub> ..	26.11%
NaCl ..	21.04 ..
Ca(OH) <sub>2</sub> ..	24.13 ..
NaHO ..	5.17 ..
Na <sub>2</sub> SO <sub>4</sub> ..	8.28 ..
SiO <sub>2</sub> ..	1.22 ..
Fe <sub>2</sub> O <sub>3</sub> +Al <sub>2</sub> O <sub>3</sub> ..	0.74 ..
OH ..	3.34 ..
Gelatinous matter ..	6.94 ..
	100.00 ..

"Anti-incrustant powder." Maker J. Ch. Schweiger, Dessau.

Na <sub>2</sub> CO <sub>3</sub> ..	14.36 ..
Na <sub>2</sub> O SiO <sub>2</sub> ..	12.50 ..
NaCl and Na <sub>2</sub> SO <sub>4</sub> ..	0.92 ..
Organic ..	14.60 ..
Fe <sub>2</sub> O <sub>3</sub> and Al <sub>2</sub> O <sub>3</sub> ..	0.75 ..
Sand ..	12.49 ..
OH <sub>2</sub> ..	14.23 ..
	99.85 ..

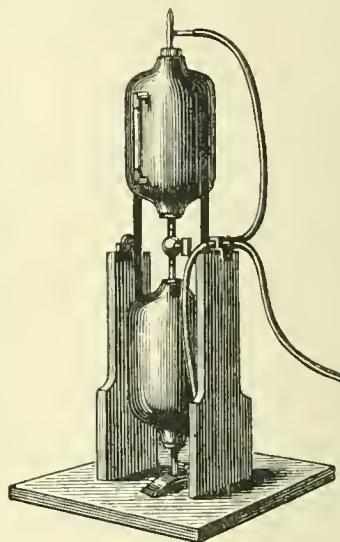
L. Cohn and Company, in Berlin, furnish the so-called "Corrosiv" of the following composition:—

NaHO ..	16.53 ..
CaO ..	3.33 ..
NaCl ..	4.71 ..
CaCO <sub>3</sub> ..	38.84 ..
Fe <sub>2</sub> O <sub>3</sub> and Al <sub>2</sub> O <sub>3</sub> ..	1.95 ..
Insoluble in HCl ..	2.71 ..
Water ..	17.89 ..
Loss on ignition ..	14.01 ..
	100.00 ..

—H. A. R.

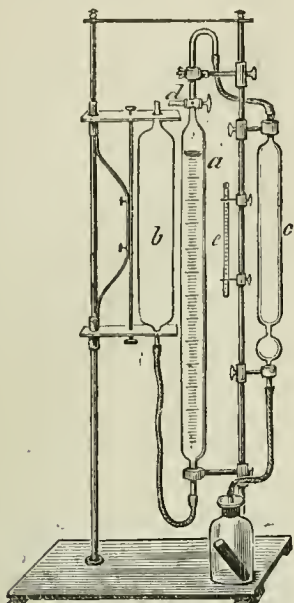
*New Apparatus for Laboratory Use.* R. Muencke, Dingler's Polyt. Journ., 65, 251, 5.

*Double Aspirator.*—This apparatus is constructed in such manner that by simply turning the aspirator occasionally round its axis it will work continuously, without necessitating the opening or closing of cocks or the changing of indiarubber tubes.



Two cylindrical vessels of equal capacity, made of sheet iron and supplied with water gauge glasses, are connected with one another in the centre by means of a brass column with an ordinary brass tap, and on the sides by two iron bars. They are closed by screws, which, on the one hand, end in bent brass tubes, corresponding to the length of the cylinders; and which on the other hand end in T shaped pieces. Diametrically to the iron bars there are screwed on axles, which move in bearings. The latter are fixed on the stand, which may be of wood or metal. One of the axles serves as plug to shell of the stop cock and the two tubes formed by one of the bearings; the other is of ordinary cylindrical shape. In order to allow the air entering the top vessel and leaving the bottom one, the plug is bored under an angle corresponding to the tubes of the shell formed by the axle; on the reverse side the plug is grooved. Its tube end is connected with the apparatus through which the aspiration is carried on, and is, by means of the angle bore, connected with one of the tubes of the axle and thereby also with the top vessel; the other tube by means of the groove in the plug arranging the communication between the bottom vessel and the atmosphere. The apparatus is kept in a vertical position by means of a spring fastened to the stand. The

tap in the brass column regulates the speed of the absorption. When the water has flown from the top cylinder into the bottom one, it is only necessary to reverse the cylinders by turning them round the axis to set the absorption going again.



*Apparatus for the Volumetric Estimation of Comparatively Large Quantities of Carbonic Acid.*—The stand consists of two strong metal bars, about 1 metre long, which are screwed on an oak plate. The levelling tube *b* moves on one bar, the measuring tube *a*, the air tube *c* and a thermometer *d* are screwed on the other so as to be movable in a vertical direction. The measuring tube, which is divided into 200 to 300 CC. and shows  $\frac{1}{2}$  CC. plainly, has at *d* a three way cock, by which communication can be effected either with the atmosphere, or with the air tube, respectively the bottle in which the carbonic acid is evolved. The tubes *a*, *b*, and *c* are connected with each other by indiarubber tubes.—J. G.

## II.—FUEL, GAS AND LIGHT.

*Improvements in Coking and in the Utilisation of the Gases from Coke Ovens.* Thos. Nicholson. Eng. Pat. 358, 1884.

THE inventor constructs or adapts ovens or kilns with interstices in and flues underneath the bottom. To the top of these ovens he adds pipes to receive the gases, and convey them through boxes partly filled with coke and gas liquor, into a main pipe connected with a condensing plant. After the tar and ammonia have been extracted, the gases are returned through another set of pipes, and used for lighting or heating purposes. To obtain illuminating, as well as heating gas, an additional range of pipes is added, the gases being collected separately. To avoid a vacuum which would create external pressure, and be liable to introduce air, the top of the oven is kept filled with steam. To produce as much ammonia as possible, and simultaneously extract the sulphur from the coke, the latter is cooled off by causing steam to pass upwards through the charge, when the process of carbonisation is completed. In order to avoid the double draught which has been found objectionable in other ovens with bottom flues, the present ovens, as well as all kilns and furnaces, are made to work from the bottom upwards, the gases being drawn off above the material. This invention can be adapted also to kilns or furnaces for burning lime or bricks, or smelting ores and similar purposes.—D.B.

*Production and Purification of Combustible Gases, and Appliances therefor.* W. S. Sutherland. Eng. Pat. 3591, 1883.

IN this invention, coal or fuel to be gasified or carbonised are placed in a producer or retort, and a substance capable of evolving hydrogen is added, which gas combines with the nascent nitrogen in the fuel to form ammonia. The latter is fixed by a suitable acid produced or given out by the decomposition of the substance employed, or by acting on the same in the producer. The author prefers the use of a soluble hydrated chloride, *e.g.* a solution of hydrated calcium chloride, although it is evident that an insoluble hydrated salt may be employed. In adding pyrites to the fuel, sulphurous acid is evolved, which fixes the ammonia passing off from the coal. A substance which is merely capable of liberating an acid may be used instead of a hydrated chloride or other salt, and the hydrogen obtained from the water in the fuel, or from the steam or water used to form water gas. In order to take up any excess of acid which may be contained in the gas after the ammoniacal salts have been collected, the gas is passed through lime or other purifiers. For the production of combustible gas, superheated steam is passed through carbonaceous fuel by combining two gas producers with a continuous superheater and a steam supply. The heated gases produced by the combustion of fuel in one of the producers, pass into a continuous superheater in one direction, and the steam from the supply being introduced into the superheater in the opposite direction, the heat is conducted through the heat-conducting surfaces separating the hot gases from the steam, into the steam from the hot gases. The hot steam is then passed through the fuel contained in the second producer, and raises it to such a temperature, that a mutual action is produced between the carbon in the fuel and the steam and carbonic oxide. Hydrogen and hydrocarbons, or water gas, are produced, and when raw fuel is used, the resulting mixture of gases is passed through suitable apparatus for separating the tar and ammoniacal products. In order to obviate the difficulty of keeping in repair the parts of the superheater into which the hot gases first enter, the fuel chamber in which the coal is burned is made of sufficient depth to produce carbonic oxide, or the well-known producer gas. The latter is then introduced into the superheater side by side with hot or cold air, so that combustion takes place inside the superheater. By this means the temperature can be controlled by properly proportioning the steam to the gases and air, and the impingement of intensely heated gases against the end walls or surfaces containing the steam, is prevented. The superheater consists of a number of tubes or suitable heat-conducting and refractory material, placed preferably parallel, and at a suitable distance apart, and in one length, or jointed up in lengths, with faucet and spigot joints, or similar means. The tubes are carried at their ends next the fireplace by a wall of refractory material, and are so kept the right distances apart, and walls are extended between the ends of the wall carrying the tubes, so as to form a chamber through which the steam passes on its way from the generator to the producer. For further details in connection with the construction of the superheater and producers, we must refer to the description of the drawings accompanying the original specification.—D.B.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Obtaining Benzene and other Hydrocarbons.* G. E. Davis. Eng. Pat. 4468, 1883.

IN a previous communication (Pat. 5717, 1882), the oil used for absorbing, and likewise the gases from the distillation of the coal, were cooled in an air cooler, or a freezing machine. It has been found that the process previously patented is also partly applicable to the gaseous products resulting from the distillation of shale, and to a certain degree to the various mixtures of gases evolved from producers, coke ovens, etc. As, however, the relatively large volume of these gases renders it



more difficult to extract the benzene from them than from those evolved in the distillation of coal, they may be treated more economically in the following manner:—The gases are compressed, an operation which results in the production of much heat. The compressed gases are cooled, after which they are brought into contact with a heavy hydrocarbon oil, to absorb the benzene and other volatile hydrocarbons. The operation of compression alone is not sufficient to absorb the whole of the benzene from the gases, these gases must therefore be allowed to expand, and during the expansion, or afterwards, as may be found convenient, the gases are passed through washers, scrubbers, or other apparatus containing the hydrocarbon absorbent.—D. B.

*Thiitolene or Methyl-thiophene.* Victor Meyer and Hans Kreis, *Berichte* 17, [6], 787.

THE authors have succeeded in obtaining this homologue of thiophene from coal-tar toluene. The method first adopted was similar to that used in the extraction of thiophene from coal-tar benzene (treatment with sulphuric acid, so as to convert into sulphonic acids, etc.), and thus a crude oil containing about 15 per cent. of thiitolene and 85 per cent. toluene was obtained. But whereas thiophene can be separated from a mixture of thiophene and benzene by repeated partial extraction with sulphuric acid, thiitolene cannot be isolated in the same manner. Meyer and Dyson have, however, shewn that by the action of iodine, in presence of iodic acid or mercuric oxide, thiophene and its homologues are converted into iodo-substitution-products of high boiling-points; and since the hydrocarbons of the benzene series are only attacked at higher temperatures by these reagents, they can be easily separated. Iodothiitolene prepared in the above manner was converted into thiitolene by the action of sodium in alcoholic solution; it is a colourless mobile oil, boiling at 113° C.

Dibromthiitolene  $C_4H_2Br_2(CH_3)_2S$  is an oil boiling at 227°–229° and which by the further action of bromine is converted into tribromthiitolene  $C_4H_2Br_3(CH_3)_2S$ .

Crude xylene from coal-tar also gives Laubenheimer's reaction, and the authors hope to isolate from this, thioxene the next higher homologue of the thiophene group.—D. E. J.

#### IV.—COLOURING MATTERS AND DYES.

*A New or Improved Colouring Matter and Process for obtaining the same.* W. P. Thompson. A communication from M. E. Savigny, of New York. Eng. Pat. 1031, January 9th, 1884.

IN this invention the wood or plant is subjected to a "fermentation" or an "oxidation" process, and is especially applicable to the treatment of tannic woods or plants. The woods or plants are reduced to a finely divided state, placed in suitable vats with tepid water sufficient to soak the wood, and brewers' yeast or other leaven added to produce "fermentation" or, a diluted acid is used to effect the "oxidation." Hydrochloric or nitric acids are suggested as best for the purpose. After the admixture of the leaven or the acid the mass is allowed to stand a considerable length of time, when more water is added, the temperature raised and kept at about 120° to 150° Fah. with frequent stirring for two days. The acid method is the quickest, requiring about one-third the time needed for the fermentation process. When the scum shows a deep orange colour the temperature is raised to the boiling point, and maintained there for an hour. The liquor is then drawn off and evaporated, resulting in the first extract. The residues left in the vat may be further treated with alkaline bodies in the same manner as heretofore; but the resulting extract is of an inferior quality. A better method for extracting the colouring matter from these residues is that described in Patent 1032, an abstract of which appears in this Journal. The inventor in using the term "oxidating" claims to cover both the "fermentation" and the "oxidation" processes.—G. H. B.

*A New or Improved Colouring Matter and Process for obtaining same.* W. P. Thompson. A communication from M. E. Savigny, New York. Eng. Pat. 1032, Jan., 1884.

THE wood or plant is first crushed, bruised, powdered or finely divided, then placed in a suitable vessel capable of being heated, and a sufficiency of water added to cover the material. The temperature is next raised to the boiling point and a quantity of a soluble body such as glycerine, or substances easily saponified by a small percentage of alkali added. The mass is then mixed and boiled for half an hour, and sufficient alkali added to saponify without excess. The clear liquid is drawn off and evaporated, the result being the first extract.

The colouring matter in the residues left in the vat is removed—after washing free from alkali—by pouring on boiling water, and while on the boil, adding one pound of commercial alum to every 100 pounds of residues, the boiling being continued until all the colouring matter is extracted. The liquor is then evaporated to get the extract. These extracts compare well with the class of colouring matters known as "Catechus."—G. H. B.

*On Diazoamidobenzene.* B. Fischer. *Ber.* xvii. [6], 641.

THE reaction between phenols and diazo-compounds, by which oxyazo-compounds are prepared, does not always proceed smoothly, and Wallach (*Ber.* xv., 28) has observed that aqueous solutions of resorcin and diazoxylenchloride do not react as long as the solution remains acid, but, on the addition of alkali, azoxylenresorcin is formed. The yield of this body is, however, far better if sodium acetate is added instead, for the presence of caustic alkalis causes the formation of insoluble bye-products. This simple but important modification, which Wallach has introduced into the method for preparing oxyazo-compounds, might be expected to succeed also in the preparation of amido-azo-compounds, but Fischer finds that such is not the case.

One molecule of sodium nitrite was added to a well cooled solution of 2 molecules aniline in dilute hydrochloric acid; on the addition of a concentrated aqueous solution of sodium acetate (2 molecules) to the clear liquid, a lemon-yellow precipitate was formed, which appeared to be amidoazobenzene. This was washed, dried, and crystallised from alcohol; on analysis it gave numbers corresponding with the formula  $C_{12}H_{11}N_3$ . On heating with concentrated hydrochloric acid, this substance split up into phenol and aniline; and when heated with alcohol and concentrated hydrochloric acid it yielded benzene and aniline. Hence it must have been diazoamidobenzene.

The author considers this to be the best way of preparing diazoamidobenzene, and remarks that this result illustrates in a striking manner the way in which the decompositions of the diazo-bodies are influenced by the presence of different reagents. Thus, if we use caustic soda instead of sodium acetate in the above reaction, amidoazobenzene is obtained directly.—D. E. J.

*On the Constitution of Phthalylechloride and Anthraquinone.* Victor Meyer. *Ber.* xvii. [6], 817.

TREADWELL and MEYER have found that phthalyl-ethyl does not, under any circumstances, combine with hydroxylamine; hence it is not a ketone, and the constitutional formula  $C_6H_4 < \begin{smallmatrix} CO \\ CO \end{smallmatrix} C_2H_5$ , usually assigned to it cannot be correct. Further, since phthalyl-ethyl is obtained by the action of zinc-ethyl upon phthalyl-chloride, the latter body must not be represented as  $C_6H_4 < \begin{smallmatrix} CO \\ CO \end{smallmatrix} Cl$ . It is

probable that both are lactones, like phthalophenone:—  

$$C_6H_4 < \begin{smallmatrix} CO \\ C \end{smallmatrix} > O \quad C_6H_4 < \begin{smallmatrix} CO \\ C \end{smallmatrix} > O \quad C_6H_4 < \begin{smallmatrix} CO \\ C \end{smallmatrix} > O$$

$$\quad \quad \quad \parallel \quad \quad \quad \parallel \quad \quad \quad \parallel$$

$$(C_2H_5)_2 \quad (C_2H_5)_2 \quad Cl_2$$
 Phthalophenone. Phthalioethyl. Phthalylechloride.  
 The lactones do not give the hydroxylamine reaction; Lach has shewn this in the case of caprolactone,

phthalide, and coumarin (Ber. xvi., 1781), and V. Meyer states that phthalophenone does not unite with hydroxylamine.

E. v. Meyer has suggested that anthraquinone is a lactone, viz., phenylene-phthalide  $C_6H_4 < \overset{\text{O}}{\underset{\text{C}}{\text{C}}} > O$  but



Dr. Goldschmidt finds that it forms with hydroxylamine the compound  $C_{14}H_9O_2N$ . This confirms the view generally held, namely, that anthraquinone is diphenylene-diketone  $C_6H_4 < \overset{\text{CO}}{\underset{\text{CO}}{\text{C}}} > C_6H_4$ .

D. E. J.

*Studies on Sulphonic Acids, No. 1. On the Hydrolysis of Sulphonic Acids, and on the Recovery of the Benzenes from their Sulphonic Acids.* Henry E. Armstrong, F.R.S., and A. K. Miller, Ph. D. (*Journ. Chem. Soc.*, April, 1884, p 148-152.

IT is well-known that several of the benzenes may be recovered from their sulphonic acids by dry distillation (Freund, *Annalen*, 120, 80; Beilstein, *ibid.*, 123, 36), but the yield is unsatisfactory. When a sulphonate is heated with hydrochloric acid under pressure, the benzene may be recovered completely (*Chem. News*, 1874, 29, 282; Ber., 1874, 7, 406). The decomposition must, however, take place in closed vessels, and hence there is risk of loss by explosion. Jacobsen has in one case (Ber., 1878, 11, 19) substituted sulphuric for hydrochloric acid—while investigating the hydrocarbons resulting from the action of dehydrating agents on camphor, the authors wished to separate 1:2:3:5 tetramethyl benzene from admixed hydrocarbons. Mesitylene and pseudocumene had been successfully separated by heating their sulphonic acids with hydrochloric acid at 100°, when the mesitylene compound was hydrolysed, the pseudocumene being recovered by heating at 130-140°. When mesitylene is dissolved in sulphuric acid it may be recovered by passing steam into the solution, and the same process was tried in the case of tetramethyl benzene, but it was found that all the benzenes could readily be recovered. The method described by the authors consists in passing steam through the solution of sulphonic acid or sulphonate in sulphuric acid, the acid solution being heated and maintained at the temperature at which hydrolysis takes place with fair rapidity. An excess of sulphuric acid should be employed, so as to retain a sufficient bulk of liquid in the retort or flask; about equal weights of sulphonate and sulphuric acid are recommended. The yield of hydrocarbon is stated to be nearly theoretical, loss only occurring through dissolution of a small quantity of hydrocarbon by the water; through incomplete separation of hydrocarbon from the water; and through evaporation of the hydrocarbon. The temperatures at which various sulphonic acids begin to decompose was studied with the following results:—

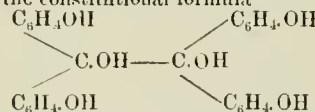
Benzene sulphonic acid .....	about 175°
Toluene para sulphonic acid .....	.. 150°
Metaxylene sulphonic acid .....	.. 120°
Orthoxylene sulphonic acid .....	.. 120°
Paraxylene sulphonic acid .....	.. 115°
Pseudocumene sulphonic acid .....	.. 106°
Mesitylene sulphonic acid .....	.. 130°
Cymene sulphonic acid .....	.. 120°
1:2:3:5 tetramethyl benzene sulphonic acid ..	.. 120°
Metamethylisopropyl benzene sulphonic acid ..	.. 120°
1:2:4 Dimethylethyl benzene sulphonic acid ..	.. 120°

Unfortunately the rate at which change takes place at the temperature of initial hydrolysis is extremely slow, and it is therefore necessary to distil at higher temperatures, so that it may happen that the hydrolysis of a more stable sulphonate may be going on slowly at the same time. Thus, when a mixture of mesitylene and pseudocumene sulphonic acids is distilled, the greater part of the mesitylene is recovered free from pseudocumene by heat to 105°, but a temperature of 105-115° is required in order to obtain the remainder, and a good deal of pseudocumene then passes over. By dissolving the mixed distillate in sulphonic acid and repeating the process, a complete separation may, however, be effected; and by combining this method with Jacobsen's, which consists in crystallising

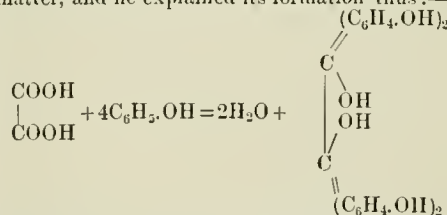
the sulphonic acids from their sulphuric acid solution, a complete purification of many hydrocarbons may readily be effected. When the sulphonates are heated in sealed tubes the temperatures of hydrolysis are not always the same as when the sulphonates are distilled. Thus mesitylene was completely recovered at 110°, while pseudocumene sulphonic acid was unacted on, and was only decomposed at about 130°. Metaxylene sulphonic acid underwent hydrolysis at 138°, while the ortho and para compounds were unacted on, the temperature for these substances being about 160°. Further researches on hydrolysis under pressure are promised, and the experiments will be extended to hydrocarbons other than benzenes.—S. Y.

*On the Colouring Matters formed by the action of Phenols on Aromatic Aldehydes.* Carl Zulkowsky. *Monatshefte für Chemie*, Bd. V. [ii and iii] 108.

LIEBERMANN and Schwarzer obtained the so-called rosolic acid in the year 1876, by the action of salicyl aldehyde on phenol, and considered with H. Fresenius that in the corallin process of Kolbe and Schmitt, the nascent carbonic oxide gave rise to rosolic acid. Baeyer and Emil Otto Fischer, on the other hand, attributed the formation of the colouring matter to nascent carbon dioxide. The author inclined to the latter view, and believed that the formation of the resinous constituent of corallin was also due to the nascent carbon dioxide. The composition of the rosolic acid, as well as the corresponding compound formed by the action of paroxybenzaldehyde on phenol, was found later by Liebermann to differ widely from that of aurin; and he then placed the compounds among the aldehyde-phenol condensation products obtained by Baeyer, explaining its formation by the equation  $2C_7H_5O_2 + 2C_6H_5O + O = C_{26}H_{22}O_6 + H_2O$ , and giving it the constitutional formula



The great resemblance of the product to corallin led him to believe that it was the chief constituent of that colouring matter, and he explained its formation thus:—



He also believed that aurin was formed from this compound by the further action of phenol and sulphuric acid, thus  $C_{26}H_{22}O_6 + 2C_6H_5OH + O = 2C_{19}H_{14}O_3 + 3H_2O$ . When corallin was dissolved in caustic soda and the solution supersaturated with sulphur dioxide a resinous substance was deposited, while aurin and methyl aurin remained in solution. These were precipitated as sulphites on addition of hydrochloric acid, heated for some time in vacuo to 122° to remove sulphur dioxide and recrystallised from 60 per cent. alcohol. The mother liquor was evaporated, the residue dissolved in absolute alcohol, and the remaining aurin precipitated with ammonia gas. The filtrate after evaporation and treatment with hydrochloric acid yielded an amorphous colouring matter, which was not further investigated.

Trzeinski recently isolated a substance from the reaction products of salicylaldehyde and phenol by means of acid sodium sulphite, which had the composition  $C_{19}H_{14}O_3$ , and to which he gave the name oxyaurin. (Berl. Ber. 16, 2835.) In his investigation of the action of salicylaldehyde on phenol, the author used much less aldehyde than either Liebermann or Trzeinski. Numerous experiments led to the conclusion that a colouring matter resembling aurin is only found when the phenol is in large excess, and that the yield is

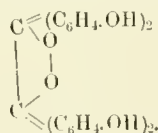


better when the mixture is heated to 120° for some time. The best results were obtained by mixing 50grms. of salicylaldehyde with 154grms. of phenol, cooling well and adding gradually a mixture of 26grms. of glacial acetic acid and 82grms. of concentrated sulphuric acid. Each drop of the acids produced a red colouration, and the mixture finally became thick and of a brown-yellow colour. On heating half a day to 120° the product attained a metallic lustre and became denser. The heated mass was poured into hot water, stirred and allowed to cool. The acid liquid was decanted off and the green resinous residue boiled with water three or four times to remove phenol. The product was then found to resemble corallin exactly. Neither aurin nor oxyaurin were obtained, but the crude product was always a mixture of two amorphous bodies, one of which could be purified. The crude substance was dissolved in moderately concentrated warm caustic soda and treated with excess of sulphur dioxide, when an ochre-yellow substance was deposited. The deep yellow filtrate was heated to 90° and precipitated with concentrated hydrochloric acid: at higher temperatures the colouring matter forms a resinous mass and is difficult to purify. The precipitate was filtered, washed free from chlorine and dried at the ordinary temperature. The pure product resembled corallin closely, but differed in many respects from aurin. Thus (1) ammonia gives no precipitate with the alcoholic solution; (2) it is difficultly soluble in glacial acetic acid; (3) the alcoholic solution has a red colour with a violet tinge; (4) it is amorphous; (5) it does not yield rosaniline or any similar body by heating to 180° with alcoholic ammonia; (6) nascent hydrogen, though it decolourises the solution, forms no crystalline leuco-compound; (7) dioxybenzophenone is not formed by heating the product with water to 250°, but only tarry matters. The quantity of the pure colouring matter is equal to that of the salicylaldehyde employed. With smaller quantities of phenol the substance is not formed, or only in minute quantities.

To purify the ochre-yellow product the process of separation was repeated, and the precipitate dissolved in alcohol and poured into water. The precipitate settled down only after warming with hydrochloric acid. The dark metallic-looking green grains form an intense brown-yellow solution with alcohol, and a very deep red violet solution with alkalis. It yields no blue colouring matter with aniline, and its properties thus agree with those of the oxidation product of the resinous constituent of corallin. The analysis of the substance shewed that it was not pure.

Paraoxybenzaldehyde and Phenol.—The same process was employed as with salicylaldehyde and the same products were apparently obtained.

The pure colouring matter resembling aurin appears to have the composition  $C_{26}H_{20}O_6$  and not  $C_{26}H_{22}O_6$  as given by Liebermann. The latter, according to the author, agrees better with Trzeinski's results. Its formation may be explained thus  $2C_6H_5O_2 + 2C_6H_5O + 2O = C_{26}H_{20}O_6 + 2H_2O$ , and Liebermann's structural formula would require to be modified as below:—



The oxygen is supposed by Liebermann to be derived from the sulphuric acid, but the author noticed no evolution of sulphur dioxide, and he is inclined to believe that the oxygen is derived from the air. He proposes the name aurin for the new colouring matter, on account of its resemblance to aurin. The compound bears very great resemblance to the substance isolated from corallin by the removal of the resinous product and the aurins, but their identity has not been fully proved. They have the following properties in common; (1) they are similar in appearance and cannot be crystallised; (2) they do not form rosaniline when heated to 180° with alcoholic ammonia; (3) they yield a blue colouring matter with aniline; (4) they are soluble in acid sodium sulphite.

The alkaline solution has, however, a yellow colour, and the composition is somewhat different. The author concludes that corallin contains a substance which is either identical with, or nearly related to aurin, and he considers it doubtful whether the aromatic aldehydes are concerned in the formation of aurin.—S. Y.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

*A New or Improved Process for Separating Wool from Sheepskins in the Fresh State.* Paul Henri Picard-Gouler fils. Eng. Pat. 4199, Aug. 31, 1884.

INSTEAD of using any of the ordinary depilatories which, according to the inventor, have an injurious effect both upon the wool and upon the skin, he effects the separation of the wool in the following way:—

The skins, freshly removed from the animal, are hung up, after having been well stretched, in a chamber hermetically sealed, and are allowed to remain there for a few hours in summer, or for one day in winter. During this time a certain amount of heat is given off, which causes the pores of the skin to open, and so far loosens the wool that it can be very readily removed by simple picking.—E. J. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Dyeing of White mixed Silk and Cotton Goods in Two colours.* Dingler's Polyt. Journ., 251., Heft 13.

IX *Bulletin de Mulhouse*, 1884, p. 65., O. Brenner describes a process long employed in Lyons for dyeing goods consisting of both silk and cotton in two colours. The silk is dyed first and in such a manner that the cotton does not take up any colouring matter, likewise the cotton is then dyed without altering the shade of the silk. This is done by means of a bath of tannin between the two dyeing operations, and as an example may be taken the dyeing of the silk rose and the cotton green. The silk is dyed first and this is done with the best and fastest colours. It is dyed warm and with a little soap, the liquor in which the silk has been boiled is very suitable after the addition of some acetic acid. Unless the latter precaution be adopted the silk will take up the colour badly and unevenly. After dyeing comes a thorough washing, and should the cotton shew a slight tinge this is removed by very dilute bleaching liquor. The most important part of the process is the tannin mordanting, and this is done as follows:—Eight per cent. of the weight of the piece in pure tannin is dissolved in water and then applied for 12 hours, dry without washing. After drying, fix with tartar emetic 150grms. to the piece (generally weighing from 2½k. to 3k.), time 2½ to 3 hours in the cold. Wash with water. Dye the cotton in the cold as quickly as possible, using sufficient colour to obtain the shade required at once, and acidify the dye-bath slightly with acetic acid. The process should not last longer than about thirty minutes, in order to keep the silk as clear as possible. After dyeing comes washing in a pretty concentrated soap bath, through which the piece is taken for from ten to fifteen minutes in order to remove any colour that the dyed silk may have taken up. If necessary soap again and wash. For rose it is best to use "Rose Bengale" or Eosin soluble in alcohol, and for the green "Vert Brilliant." By means of this process beautiful effects and the most miscellaneous grouping of shades can be obtained, and it has the advantage over the older process of weaving the dyed yarn, that it makes the manufacturer more independent of rapidly changing fashions. It ensures the manufacturer against the depreciation in value of his goods since he only needs to dye as much as is requisite to supply orders in hand.—II. A. R.

## VII.—ACIDS, ALKALIS, AND SALTS.

*A New Method for Treating the Residues from Soda Works.* G. F. Redfern, London. A communication from Dr. H. von Miller and Carl Opl of Hruschau. Eng. Pat. 2334, Jan. 29, 1884.

ALKALI Waste is converted by any known method into soluble calcium hydrosulphide, and the solution of this latter is poured into a suitable vessel provided with a stirrer, and heated either by a furnace or with high pressure steam. When boiling point is reached decomposition ensues, calcium hydrate being precipitated and sulphuretted hydrogen being disengaged. Half of the latter is used for obtaining sulphur or sulphuric acid therefrom, and the other half being allowed to act upon a quantity of the waste suspended in water is employed in the preparation of a further supply of calcium hydrosulphide.—A. R. D.

*Improvements in the Manufacture of Carbonic Acid Gas.* Gustav Jarmay, Winnington, Eng. Pat. 4415, Sept. 14, 1883.

WATER Gas, well washed and scrubbed, is burned with as nearly as possible the exact quantity of air required for its combustion, in a suitable kiln packed with limestone or other earthy carbonate. As the water gas is of great heating power, and comparatively very little of it is required for the calcination of the limestone, the exit gaseous product is extremely rich in carbonic acid, and yields more of that gas in proportion to its bulk than the mixtures obtained by other methods now commonly in vogue. Moreover, it is free from tar and volatile matters, and the lime obtained is free from cinders.—A. R. D.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*On the Detection of adulterations in Portland Cement.* R. Fresenius and W. Fresenius. Zeitschrift für Analytische Chemie, 23, 175.

THE experiments described in this paper were undertaken at the request of the German Association of Cement Manufacturers, on account of the increasing adulteration of Cements. Numerous substances such as ground shale, limestone, and so on, are readily detected, and are, therefore, not so often employed as formerly, but other substances of nearly the same composition as Portland cement have taken their place. Experiments were made with 12 samples of pure Portland cement from different parts of Germany, England, and France, and these were compared with three kinds of hydraulic lime, three of weathered slag-dust, and two of ground slag. The cements were of various ages and had been exposed to the air for different lengths of time. There appeared to be in many respects a greater difference in the behaviour of the various pure Portland cements than between these and the adulterants, but the following determinations are sufficient to distinguish between them, and to detect the presence of the adulterants in Portland cement:—(a) the specific gravity, (b) the loss on ignition, (c) the alkalinity of the water in which the substance has been placed, and the action (d) of dilute acid, (e) of potassium permanganate, and (f) of gaseous carbonic anhydride.

(a) For the specific gravity the method of Dr. Schumann was employed. The apparatus consists of a glass flask of 100-150c.c. capacity, in the very short neck of which is placed like a stopper a tube of 40c.c. capacity graduated in  $\frac{1}{10}$ c.c. The vessel is filled to the zero point with turpentine, and a weighed quantity of the solid introduced gradually by means of a funnel. The weight divided by the increase of volume gives the specific gravity. The end of the graduated tube should be loosely corked to prevent evaporation, the temperature should be kept constant, and the vessel should be carefully shaken to remove all air from the solid. The specific gravity of pure Portland cement is never lower than 3.1, while that of the adulterant examined is always

lower. The results were the same when the cement was very finely ground.

(b) About 2grms. were heated in a platinum crucible over a Bunsen burner for 20 minutes. The difference between Portland cement and hydraulic lime is very considerable.

(c) The substance was finely powdered and passed through a sieve of 5000 meshes to the square centimetre. One gm. was shaken with 100c.c. of distilled water at the ordinary temperature for 10 minutes. The solution was passed through a dry filter and 50c.c. titrated with decinormal hydrochloric acid. The difference between the cement and the hydraulic lime is in this case considerable, that between the cement and the slag less so. The results with the lime are highest, and with the slag the lowest.

(d) One gm. of the ground substance, of the same fineness as in (c), was shaken with 30c.c. of normal hydrochloric acid and 70c.c. of water for 10 minutes. 50c.c. of the filtrate were titrated with normal caustic soda. The results with slag are considerably lower than with the other substances.

(e) The determination of the sulphur present in the form of metallic sulphides always came out lower in a mixture of cement and slag than when the two were analysed separately, because in the cement the iron was present as ferrie, and in the slag as ferrous oxide. The action of permanganate was therefore studied. One gm. of the finely powdered substance was treated with 150c.c. of a mixture of one part of dilute sulphuric acid (sp. gr. 1.12) and two parts of water, and the solution was titrated with potassium permanganate. The numbers with slag were very much higher than with cement or lime, and were equally reliable with the slag alone, or with a mixture of slag and cement. Hence the quantity of slag in the mixture can be determined.

(f) About 3grms. of the finely powdered substance (as in c and d) were placed in a weighed tube and subjected to the action of carbonic anhydride, dried over sulphuric acid for about two hours, or until the weight became constant. The increase in weight gave the amount of carbonic anhydride absorbed. A small calcium chloride tube was placed at the end of the weighed tube to absorb any water that might be evolved. The hydraulic lime was found to absorb by far the largest quantity of the gas. The numbers in the case of a pure Portland cement should be as follows: (a) Specific gravity, not lower than 3.1; (b) loss on ignition, between 0.3 and 2.59 per cent., or at least not much higher; (c) alkalinity of aqueous solution of 0.5grms. of cement, corresponding to 4 to 6.25c.c. of decinormal acid; (d) volume of normal acid neutralised by 1gm. of the powdered cement, 18.80 to 21.67c.c., at any rate not much lower than 18.8; (e) weight of permanganate reduced by 1gm. of cement, 0.79 to 2.80m.grm., or not much more than 2.8; (f) weight of carbonic anhydride absorbed by 3grms. of cement 0 to 1.8m.grm. The tests a, c, d and e are applicable for the detection of slag, and a, b, c and f for the detection of hydraulic lime.

Experiments were made by mixing nine parts of pure cement with one part of hydraulic lime and with one part of slag respectively, and it was found that the adulteration could in every case be detected by the method described.

Of course other substances may be employed to adulterate the cement, but the purity of the substance may probably be considered to be proved whenever the numbers fall within the limits given above.—S. Y.

## X.—METALLURGY, MINING, Etc.

*Modern Progress in Mine Engineering.* An Address delivered to the Liverpool Engineering Society, at the Annual Meeting, December 5, 1883, by Henry Bramall, M. Inst. C.E.

THE first operation here treated of is boring, which, since the difficulties occasioned by the breaking of the



iron rods carrying the chisel were removed by the invention of the free-falling tool, has been brought to a high degree of perfection. By a method much in vogue in the Pennsylvania oil district, where "the chisel is attached to a short length of rods, with jars, and these are connected by a round hemp rope, with a reciprocating beam worked by a steam engine," 39ft. have been bored in 24 hours at a cost of  $\text{Ss. } 5\text{d.}$  per foot for a 5in. or 6in. hole. By Mather and Platt's machine, in which the boring head is connected with the motor by a flat wire rope, cores of the strata may be obtained from holes of from 9in. to 18in. diameter. The speed attained is from 4ft. to 9ft. per 24 hours. Another important invention is the diamond drill, proposed by Heman in 1854, patented by Leschot in 1862, and since improved by Col. Beaumont. Diamonds of the black or carbonado variety are mounted by pressure at the end of a steel tube, which, being rotated, cuts an annular groove in the rock. A stream of water, passing down the inside of the tube and up again between this and the sides of the hole, keeps the cutting surfaces clean and brings away the *débris*. At Mecklenburg, by this machine, a depth of 4000ft. is said to have been reached in six months. One of its chief recommendations is the perfection of the cores obtained, while on the other hand it appears to cost from 50 per cent. to 75 per cent. more than rigid rods, and there is an increased liability of the holes to become crooked. In the Aalborg system, invented by Mortensen, holes are bored through alluvium without any cutting tool at all. Two tubes are sunk, an inner and an outer, and water, being forced down the former, ascends by the annular space between it and the latter, bringing up the *débris*. From 60ft. to 100ft. have thus been sunk in a day. Drills driven by steam or compressed air have to a great extent superseded hand-labour for drilling holes for blasting. Most of them are upon the percussive principle, the drill being attached to the end of a piston rod. In Brandt's machine, which is said to be the most effective and economical, the drill is a hollow cylindrical steel bar, which is forced against the face of the rock by a hydraulic press, and rotated by a pair of small hydraulic engines. The core of the cylindrical hole thus cut is washed away by the continuous stream of water escaping from the driving cylinders. The tunnelling machines of Colonel Beaumont and Mr. Brunton grind away the whole face of a 7ft. heading at one operation by a series of cutters carried on a rotating boring head. Among other quite modern innovations is the use of strong explosives. Dynamite was introduced in 1864 by Nobel, who has since invented blasting gelatine, which is prepared by dissolving collodion cotton in nitro-glycerine. This is recommended by its insensibility to shocks and the absence of dust after explosion. A recent invention for coal getting takes advantage of the fact that caustic lime undergoes a great increase of bulk on the addition of water, but the results from its use are somewhat uncertain, and neither this nor the many varieties of wedges that have been from time to time introduced have been able to displace gunpowder. The use of coal-cutting machines, from the plant and the necessity of a good situation entailed, has been found to be advantageous chiefly in the case of a thin, hard seam. Some, as Winstanley's, Rigg's and Meiklejohn's, cut out a groove after the manner of a circular saw. In Baird's the cutters are fixed upon a travelling pitch chain, while in the Lechner, an American machine, they are mounted on a revolving bar driven by pitch-chains. Within the last few years a great increase has been attained in England in the depths of shafts. The deepest, Ashton Moss, is 900 yards. The speed in sinking them has been increased, too, by means of a better organisation of labour. An American plan of drilling a series of holes with a diamond drill, and blasting them away in sections, has met with considerable success, as it saves refixing the drilling tackle after each blast. Shafts are generally lined with brickwork, but at Saarbruck iron rings have been used at intervals of 39 inches with a lining of 2in. oak planks. In another case wrought iron plates have been substituted for the planks. The greatest difficulty to be met with in shaft sinking is

caused by the presence of water. Many ingenious plans for overcoming this are due to our Continental neighbours. The Deutscher Kaiser pit was sunk through 244ft. of alluvium and 76ft. of cretaceous marls (without withdrawing the water from the shaft) by a rotating boring tube filled with leathern bags for removing the material, the cast-iron lining cylinders following the tool in its downward progress. The diameter was 17ft. at the top and 13ft. 4in. at the bottom, and the whole work was completed in 20 months. The Kind Chaudron system, in which the boring is effected by the percussive action of a series of steel cutters carried on a *trepan* or boring bar, is useful in the case of ground which, while moderately firm, contains heavy feeders of water. The boring bar weighs 20 tons, and the bucket for removing *débris* has a capacity of 17 tons. M. Poetsch deals with loose saturated alluvium by freezing it. A concentrated solution of the chlorides of magnesium and calcium at a temperature of about  $13^{\circ}$  below zero is circulated in copper pipes through the mass which, when frozen, is dealt with as a solid. Of late years the increased depth of shafts has necessitated winding engines of extraordinary size and power. At Harris's Navigation the cylinders are 54 inches in diameter, and capable of lifting a load (including rope, cage, &c.) of  $15\frac{1}{2}$  tons at the rate of 32 feet per second. To compensate for the weight of the winding rope, which in deep pits amounts to 25 per cent. or 30 per cent. of the whole load, a conical drum with a spiral groove for the rope to lie in is in use in most large winding plants. At Clifton Colliery, near Manchester, ropes coiling upon themselves in a vertical iron groove between two iron discs are giving satisfaction. Round wire ropes are fast replacing those of hemp. To remedy the evil of the labourers in metalliferous mines having to ascend and descend by means of ladders the "Man Engine" was invented. It consists of a reciprocating rod or rods having platforms attached, on or off which the men step in their progress up and down. According to the Prussian report for 1881, the deaths per 1000 where it is used are 0.383, compared with 0.084 where ladders are still in vogue. The pneumatic system of raising a load has been practically applied in France. A wrought-iron tube, 5ft. 3in. in diameter, is fitted with a piston cage, which, when the air above it is exhausted, rises with a load of three tons at the rate of  $19\frac{1}{2}$  inches per second. Underground tramways will be seen to be capable of improvement, when we find that the fractional resistance is from 35lb. to 60lb. per ton against 8lb. per ton on railways. In most mines power has to be transmitted for underground purposes, and a convenient—if not a very economical—way of doing this is by means of compressed air. Electricity has also been tried, but it introduces a dangerous element, and yields a low percentage of result. For haulage underground, endless ropes or chains are found to give the best results, although compressed air engines and (in non-fiery mines) electrical locomotives have been giving satisfaction. The old Cornish pumping engine still does duty for unwatering mines, but in draining dips, direct acting pumps, actuated by hydraulic power, are of great service. Though fans were in use for producing currents of air three centuries ago, it is only in recent years that they have been employed for ventilation of a size and power commensurate with modern collieries. The best fans are decidedly more economical than furnaces, and infinitely safer. Modern progress in mining is shewn most conclusively by the fact that 30 years ago deaths from accidents were more than twice as numerous as now in proportion to the number of people employed.—A. R. D.

*On the Detection and Estimation of Arsenic, Sulphur, Phosphorus and certain Metals, occurring in small quantities in Commercial Native Copper.*—O. Kuhn. *Zeitschrift für Analytische Chemie.* 23, 165.

THE author criticises a paper by Dr. Löwe (*Zeitschr.* 21, 516) bearing the same title, and proposes various modifications of the method there described.

According to the author, it is only when large quantities of both lead and sulphur are present that lead sulphate is contained in the precipitate insoluble in nitric acid. The whole of the lead sulphate is only precipitated on addition of alcohol. The precipitation of the arsenic from the ammoniacal copper solution is objected to on account of the partial solubility of magnesium arsenate, and the large quantity of water which must be present; it is stated that even alcohol does not cause the precipitation to be complete. The same objection is said to apply, though to a less degree, to the precipitation of phosphorus by the same method. The separation of zinc, nickel and cobalt, by precipitating the copper as sulphide, is considered unsatisfactory on account of the large quantity of copper sulphide which must be washed, and the facility with which it is oxidised.

The author takes three separate portions of copper. The first (0.5 to 0.8 grms.) is dissolved in nitric acid, and the copper estimated by titration, according to Volhard's method, with potassium sulphocyanide. In the second portion (15 to 18 grms.) the silver, lead, bismuth (iron and manganese), are estimated; and in the third (15 to 18 grms.) the arsenic, phosphorus, sulphur, zinc, nickel, cobalt, iron and manganese. Both the larger portions are dissolved separately in nitric acid, evaporated to dryness, and the residue dissolved in water, with addition of just sufficient nitric acid. The solution is filtered, and the residue washed with hot water, and the antimony, tin, silicon, phosphorus, arsenic and lead sulphate, if any, estimated. If the stannic oxide is coloured with iron, it must be fused with sodium carbonate and sulphur.

In the first portion the silver is precipitated with hydrochloric acid, and the lead with sulphuric acid and alcohol. Up to this point sufficient acid must be present to keep the bismuth in solution. The filtrate from the lead is supersaturated with ammonia to precipitate the bismuth and iron. If much phosphorus is present, the precipitation of iron is incomplete, and both the precipitate and filtrate contain iron and phosphorus. If this is so, the magnesium phosphate, produced by addition of magnesium mixture to the filtrate, will be yellow. It is, therefore, better to estimate the iron in the second portion.

The solution of the second portion is treated with barium nitrate to precipitate the sulphur, and evaporated to dryness, with sufficient sulphuric acid to convert the whole of the metals into sulphates. The residue is dissolved in water, treated with sodium carbonate until a permanent precipitate is just formed, heated to near the boiling point with sulphurous acid, and the copper precipitated with potassium sulphocyanide. After cooling, the liquid, with the precipitate, is transferred to a flask of 500 c.c. capacity, filled with water to the mark, and a further quantity of water equal in volume to that of the precipitate added (sp. gr. of the precipitate is 3.16, and 10 grms. of copper give 19.16 of copper sulphocyanide = 6.06 c.c.). The whole is then well shaken. 400 c.c. of the solution are then passed through a dry filter, evaporated to a small bulk, and treated while hot drop by drop with nitric acid, until the sulphocyanide is decomposed, and the hydrocyanic acid removed. The liquid is boiled with sulphurous acid, the excess removed by boiling, and the arsenic precipitated from the warm solution with sulphuretted hydrogen. The iron, nickel, cobalt, zinc, and manganese, are then precipitated by addition of ammonia and ammonium sulphide, and estimated in the usual manner. The ammonium sulphide in the filtrate is decomposed with hydrochloric acid, and the phosphorus precipitated as magnesium phosphate. On account of the large amount of potassium salts, the precipitate should be dissolved in hydrochloric acid, and precipitated again with ammonia and magnesium mixture.—S. Y.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*Improvements in Apparatus and Method for the Extraction and Refining of Fatty Matters from Bones, etc.* A. C. Henderson. A communication from Dr. W. Schneider, of Bavaria. Eng. Pat. 4027, August, 1883.

The apparatus consists of a boiler in which the bones, etc., are placed. Into this boiler some solvent liquor, as

for example, benzene is conducted from a reservoir by means of a tube communicating with the bottom of the boiler. Above this pipe is a serpentine steam-pipe used to bring the liquid to the boiling point. The vapour of the benzene rises through the bones, etc., to the lid of the boiler, entering a tube connected with a condensing pipe. So soon as the benzene vapour reaches the boiler lid the steam is shut off, and two taps are opened, allowing the solution of fats to run into a second boiler, situated below the level of boiler, and also fitted with a steam pipe at the bottom. After a sufficient quantity of fat solution has run into boiler, steam is turned on and the benzene vapour rises through the lid and into the base of boiler, with which it is connected by a pipe, passing through the bones, etc., to the condensing tube mentioned above, and is received in suitable reservoirs in connection. From these reservoirs the benzene is forced up pipes to the top of boiler, and enters long perforated tubes placed horizontally and vertically in the boiler; passing downwards it carries with it all fats into the boiler. This operation is a continuous one. After all the fats are removed from boiler, steam is introduced at the bottom of boiler, and passes through boiler, driving all remaining benzene into the condensing apparatus, to be used again as before.—G. H. B.

## XIV.—AGRICULTURE, MANURES, Etc.

*Occurrence of Phenol in Pinus Sylvestris.*—A. B. Griffiths, Chem. News, 49, 95.

By digestion with warm water phenol can be extracted from the stem, leaves and cones, and its presence shewn by the usual reactions. Quantitative determinations shewed that the older parts of the stems contained 0.1221, younger parts, 0.0654 per cent.; the leaves, 0.0936 to 0.0315, cones, 0.0773 to 0.0293 per cent.—D. E. J.

## XV.—SUGAR, GUMS, STARCHES, Etc.

*Improvements in the Manufacture of Glucose.*—Leon Barbier, 15 Rue des Halles, Paris. Eng. Pat. 3964, Feb. 26, 1884.

THE object of this invention is the simplification of the manufacture of glucose, and the process consists in the separation of the albumenoid and nitrogenous matters from the juice proceeding from the direct and complete saccharification of vegetable substances, by the means employed in the manufacture of crystallisable sugar, viz., by the operation known as double carbonation.—W. M.

*Improvements in Filters for Saccharine Juices, Syrups, and other Liquids.* Chas. D. Abel, London. A communication from Alfred Perret, of Roze, Somme, France. Eng. Pat. 3892, Aug. 11, 1883. Provisional protection granted only.

"THE present invention relates to the use for filtering liquids, more particularly saccharine juices or syrups, of sponges or spongy materials, or materials such as loose textile fabrics, cotton and other vegetable fibre, pumice stone, asbestos, hair, spun glass, fibrous metal, and the like, which will retain the solid impurities contained in liquids passing through them, by molecular or capillary attraction and by simple adhesion, instead of solely by the ordinary filtering action such as heretofore employed, where the filtering medium is of such close texture as to prevent the impurities passing through."

For this purpose a long horizontal vessel of rectangular transverse section is divided by loose perforated partitions into a number of compartments. Each partition is attached to a transverse bar at top, resting with its ends on endless pitch chains on each side of the vat, passing round pitch wheels at the ends, by the turning of which motion can be imparted to the chains and bars. The compartments between the partitions are filled with



the spongy or fibrous filtering material, and the liquid to be filtered is made to flow slowly through the several compartments, whereby the greater portion of the solid impurities is retained in the first compartments. When the filtering material in the first compartment has become sufficiently charged with impurities, the flow of liquid is stopped, and the filtering material with the corresponding partition is removed. All the partitions with their filtering material are now moved one step forward by means of the endless chains, and the filtering material of the first compartment, after having been freed from the accumulated deposit, is placed with its partition in the last compartment and the filtering process proceeded with as before.—W. M.

*Improvements in the Manufacture of Sugar and in Apparatus therefor.* W. R. Lake, London. A communication from Konrad Trobach, Berlin. Eng. Pat. 3244, Feb. 13, 1884.

THE object of this invention is to produce sugar directly from saccharine substances by means of an improved process without resorting to the numerous intermediate processes heretofore employed; and it consists partly in freeing the saccharine substances from their water by means of alcohol-steam, and then subjecting the substances thus freed from their water to the action of hot liquid alcohol, in order to extract therefrom the sugar only, the nitrogenous and saline matters being left undissolved; the residue is thus valuable as food for cattle. The process also consists in passing the alcoholic liquor, or saccharine solution, through a suitable filter to neutralize such acid salts as may be contained therein before separating the alcohol from the sugar. The saccharine substances, such as shavings of beet-root or sugar-cane, are placed in a suitable vessel and exposed to the action of alcohol steam, which, passing through the saccharine material, removes the water. The alcohol steam, charged with water, is now led to a suitable rectifying apparatus, and is passed through a filter containing lime or chalk so as to neutralize any volatile acid which the alcohol steam may have taken up. After being condensed, the alcohol is again used to free the saccharine material from water. The water-free saccharine substance is mixed with hot alcohol, and the alcoholic solution of sugar is run into another suitable heated vessel. In this vessel the alcohol is driven off and condensed, the sugar is left behind, and the process is continued until all the sugar has been extracted. The alcoholic solution of sugar is passed through a filter composed of chalk or lime before it is run into the vessel in which the sugar is separated from the alcohol, in order to neutralize such acid salts as may be contained in the alcoholic liquor. The invention also includes the special apparatus of which drawings, and a minute description, are given in the original specification.—W. M.

## XVI.—BREWING, WINES, SPIRITS, Etc.

*An Improved Method or Process of and Apparatus for Extracting Essential Oils from Hops or other Plants.* William Robert Lake. A communication from Théophile Auguste Breithaupt, Strasbourg, Alsace.

THE hops are placed in a cylindrical copper vessel provided externally with a sheet metal casing, and closed at the top by a cover communicating by means of a cock with a spiral condensing tube surrounded with cold water. The tube is connected with a cylindrical vessel similar to that which contains the hops, and in which a vacuum may be formed by means of a pump, with which it communicates by a pipe provided with a cock. The first vessel has a perforated false bottom on which the hops are placed, and below which are two pipes, one for the admission of steam, the other of compressed air. A third pipe serves to introduce steam into the sheet metal casing. A small vessel is connected with the lower part of the second cylinder to collect the oil, and is so arranged that it can be closed hermetically.

The first vessel is filled with hops and is heated by the steam in the metal casing. A little steam is then admitted into the vessel, and the whole is kept at about

80° by means of the steam jacket. Air is then forced into the vessel by the pump or some other arrangement until the pressure rises to about two atmospheres, and when it is saturated with the vapour of the essential oil, it is allowed to pass through the condensing tube into the vacuum cylinder. In this way the greater part of the oil is condensed, and is collected in the vessel below the second cylinder.—S. Y.

## XIX.—PAPER, PASTEBOARD, Etc.

*Manufacture of Artificial Ivory.* Henry Harris Lake, of the firm of Haseltine Lake and Co. Patent Agents, Southampton Buildings, London. A communication from Jarvis Bonestell Edson, Massachusetts, U.S. Eng. Pat. 3930, Aug. 14, 1883.

ACCORDING to the present invention xylonite, or its equivalent substance, being a cellulose compound, and consisting chiefly of pyroxyline is cut into sheets of different densities, sometimes also of slight differences in colour and combined by rolling and applying great pressure into one entire mass, but preserving the peculiar characteristics of the structure of each sheet. When this compound is cut into thin sheets, the various sections in the said sheets will shew the grain of the original sheets of which the whole is composed and so imitate the laminated character of different growths of ivory.—H. A. R.

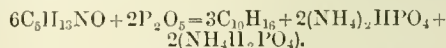
*Improvements in Paper Manufacture and in Machinery connected therewith.* Joseph Dixon, Oughtybridge, York. Eng. Pat. 4422, Sep. 15, 1883. Provisional protection only.

THIS invention relates to improving the "felting" power of the pulp fibres to an extent hitherto only attained in hand-made paper. The appearance of the paper is also to be improved as regards finish. A suitable mouth-piece or several of them formed like a tube are supported closely over the wet pulp and extend across the wires from side to side of the deckle frame, these pipes being connected with a fan or blower. When the pulp flows on to the machine wire from under the slice, the fibres are drawn parallel with the direction of motion of the machine wire. This effect is slightly rectified by the shake, but the action of the stream of air impelled against it felts the pulp better and more closely.—H. A. R.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

*Artificial Formation of a Terpene.* Radziszewski and Schramm. Berichte xviii. [6], 838.

THE authors have prepared a substance having the composition of a terpene  $C_{10}H_{16}$  from common amylene. By direct addition of hypochlorous acid, amylene is converted into the corresponding chlorhydrin, which, when heated with aqueous ammonia in sealed tubes yields oxyisoamylamine  $C_5H_{11}O \cdot NH_2$ , an oily, strongly alkaline liquid. This is gradually mixed with phosphorous pentoxide and distilled, when the following reaction occurs:



The distillate is free from nitrogen; the portion passing over between 155° and 165° has the composition  $C_{10}H_{16}$ , smells strongly like turpentine-oil, and, like the latter, absorbs oxygen rapidly from the air.—D. E. J.

*The Preparation of New Soluble Neutral Salts of Cerium for Medicinal Purposes.* John Brunt Mackey. Eng. Pat. 5328, March 22nd, 1884.

CERIUM oxalate was used by Sir James Simpson about the year 1859 in cases of sickness, as a nerve tonic and for other purposes, but it had the disadvantage of being

insoluble. The nitrate and sulphate of cerium are soluble, but are too acid and astringent. The patent is for the preparation of the double citrates, tartrates, and lactates of cerium, and either potassium, sodium, or ammonium; to which is added tincture of nux-vomica, hydrocyanic acid, pepsine, chloric ether, tincture of opium, chloroform, morphia, or strychnine.—S. Y.

*Derivatives of Quinoline and Pyridine.* O. Fischer and E. Renouf. Ber. xvii. [6], 755.

1. Orthoquinoline sulphonic acid yields on oxidation quinolinic acid, and is, indeed, the best starting point for the preparation of the latter. 40grms. of the sulphonic acid yielded, on oxidation with alkaline permanganate, 23grms. of the pure pyridine-dicarboxylic acid. [The compounds here called ortho- are those which were formerly known as  $\alpha$ -compounds.]

the crystalline precipitate and re-crystallising it from dilute alcohol, ether, or ligroin. It crystallises in colourless monoclinic prisms, which melt at  $76^{\circ}$ ; the melted mass becomes crystalline again on cooling. In small quantities it can be distilled unchanged; its vapour smells like ethylamine. In alkaline solutions it is easily oxidised; when a sample is dissolved in caustic soda and shaken up with air, it is decomposed in a few minutes, black flakes being deposited. The following reactions are characteristic:—

(1.) An alcoholic solution is coloured dark brown by a trace of ferric chloride; ferrous sulphate throws down dark green flakes. The small quantity of iron contained in common filter paper is sufficient to turn colourless crystals of the kairine base violet; by longer contact they become coloured dark brown.

(2.)  $K_2FeCy_6$  gives in acid solution a voluminous precipitate, difficultly soluble in water.

(3.) Phospho-tungstic acid produces a light yellow

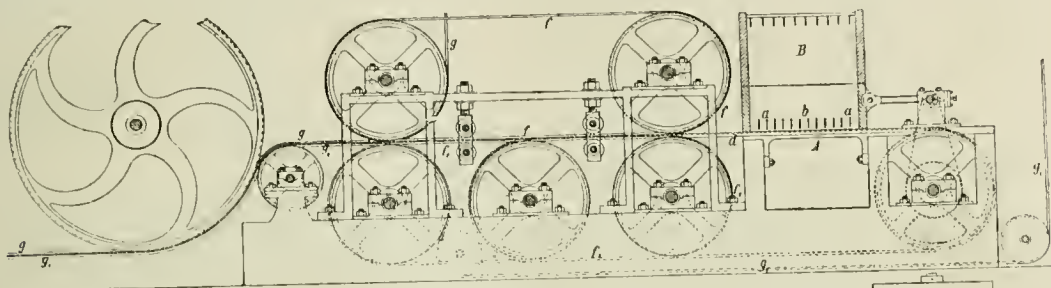


Fig. 1.

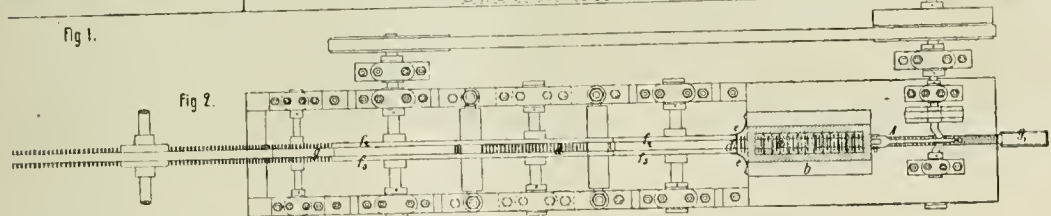


Fig. 2.

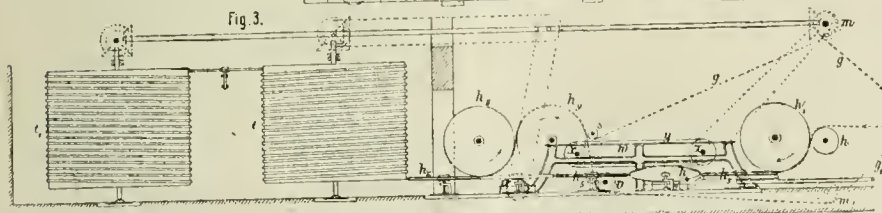


Fig. 3.

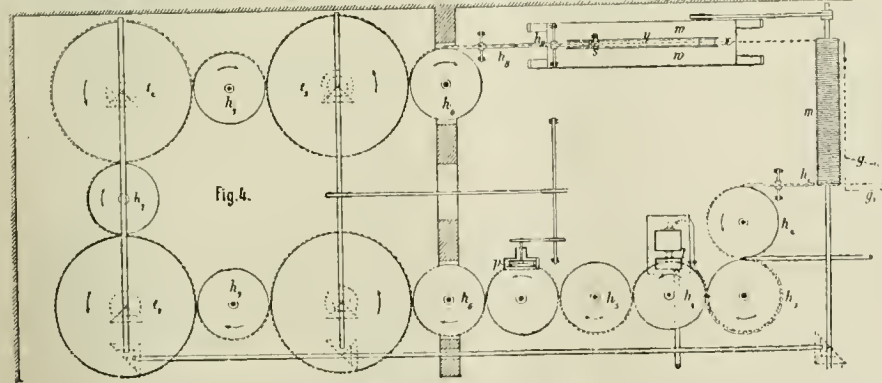


Fig. 4.

Apparatus for the Manufacture of Matches. (See Explosives, &c., page 378.)

II. Orthooxyquinoline yields the same oxidation-product.

III. Orthooxyhydroethylquinoline. The hydrochloride of this base is the artificial alkaloid known as "kairine A," and prepared in a state of great purity at the Höchst Colour Works. The free base is obtained by decomposing the aqueous solution of kairine with soda, filtering off

precipitate, which, on boiling, is converted into a dark yellow resinous mass.

(4.) On adding sodium nitrite to an acid solution a yellow colouring matter is obtained.

Kairine (orthoxyhydroethylquinoline hydrochloride). Kairine is easily soluble in water, difficultly soluble in HCl. Its taste is at first cooling, like that of saltpetre,



but afterwards bitter. An aqueous solution gives, with a little  $\text{FeCl}_3$ , a violet colour which quickly disappears; more  $\text{FeCl}_3$  produces a deep brown colour, or in concentrated solutions a dark brown precipitate. The behaviour of potassium bichromate is very characteristic; a dilute neutral solution in water becomes dark in colour on addition of  $\text{K}_2\text{Cr}_2\text{O}_7$ , and soon deposits a dark violet dye stuff, which dissolves in alcohol, yielding a mauve coloured solution.

Potassium ferrocyanide, when boiled with an aqueous solution of kairine, gives a dark yellowish green colouration.

IV. Pyridine sulphonic acids. Pyridine is not entirely converted into sulphonic acids even after boiling for 20 days with strong or fuming sulphuric acid; at least 50 per cent. remains unaltered.

$\beta$ -oxypyridine is obtained by mixing the sulphonic acid with twice its weight of caustic potash, adding a little water, and fusing carefully in a silver basin. The melt is neutralised with  $\text{HCl}$  and extracted with ether; the yield of oxypyridine is almost quantitative. It is easily soluble in water and alcohol; and forms yellowish crystals which melt at  $123.5^\circ$ .—D. E. J.

## XXI.—EXPLOSIVES, MATCHES, Etc.

*Arlow's Plant for Manufacturing Matches.* Dingler [251], Heft 12.

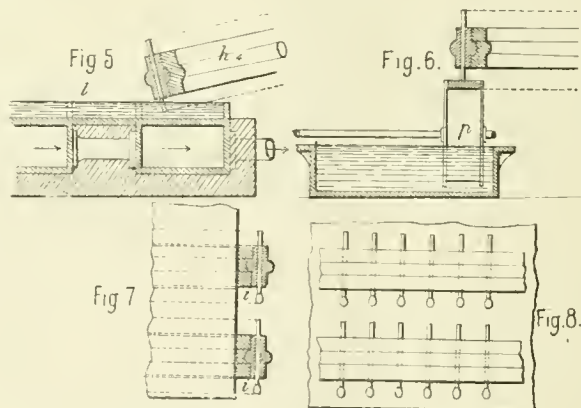
H. ARLOW in Neupst (Germ. Pat. Class 78, No. 22,308, Aug. 19, 1882) employs two endless bands which take up the matches at regular intervals, and introduce them into the different parts of the machine. By this means they are warmed, coated with paraffin, dipped, dried, and finally dropped. The matches are placed in the box B (Figs. 1 and 2) and the hopper *b* underneath is agitated by an eccentric in the usual manner, so as to introduce the matches into the grooves of the strap *d*. The width

and then over the horizontal discs  $h_2$  and  $h_3$ . A gutter heated by steam passes under the rim of the latter, so that the ends of the matches are warmed as they pass through. They then go to the inclined disc  $h_4$ , which is coated with paraffin by the bath *l* (Fig. 5) which is heated by steam, and pass as coated matches over the disc  $h_5$ , with their coated ends over the roller *p* of the dipping apparatus (Fig. 6). This is an iron box fitted with the tipping mass. The roller is covered with india-rubber, and furnished with a doctor arrangement to regulate the size of the match-head tips. After being dipped, the matches pass on over the disc  $h_6$  into the isolated drying rooms *t* to  $t_3$ . These consist of tin plate, and contain the screw motions *i* (Figs. 7 and 8). The straps pass by the disc  $h_7$  from one cylinder to the other, and finally over the discs  $h_8$ ,  $h_9$ , and  $h_{10}$ , to the place where they are deposited. The cylinders are driven by cog-wheels, and the drying is done by hot air. The arrangement for depositing the matches consists of an iron support with a level *w* on which the axle of disc  $h_9$  runs. Here two straps, *g* and  $g_1$ , separate, *g* running over the roller *s* to the spiral *m*,  $g_1$  runs over roller *v* to  $m_1$ . Two endless bands *y* run over the discs *x*, and take between them  $g_1$ , as well as the ready matches. The speed of the straps *y* is somewhat less, and causes an accumulation of matches at the end of the table *w*. Here the matches are packed into boxes.—H. A. R.

*Process for providing pieces of Compressed Gun-Cotton, Compressed Nitrated Wood, or other Cellulose, with a Coating by Treatment with a Medium Acting as a Solvent of or to the Same.* F. C. Glaser. Communicated by W. F. Wolf, of Walsrode and Max von Förster, of Berlin. Eng. Pat. 3866, Aug. 9, 1883. Provisional protection only.

"If pieces of compressed gun-cotton, compressed nitrated wood, or other cellulose, are laid or steeped in liquids which dissolve them (acetic ether, nitro benzole,

### Apparatus for the Manufacture of Matches.



of the strap and the depth of the grooves are so adjusted that their whole space is taken up by the matches which project on each side of the strap. This runs over a guide in its passage under the hopper, and this guide is furnished with small rollers (not represented in the cut) to facilitate the passage of the strap. The walls *a* in the hopper maintain the matches in their respective places, so that each groove of the passing strap contains a match. The strap *d* leaves the hopper and takes the matches along these in its grooves, and on leaving the box are straightened by two springs *c* with respect to their position on the strap. The two ends of the matches are caught by the four straps *f* to  $f_3$ , lifted out of the groove of the strap, and finally transferred to the two endless sheets *g* and  $g_1$ . Their tension is regulated automatically. The higher sheet *g* comes from the superior spiral *m* (Figs. 3 and 4), the lower sheet  $g_1$  from the lower spiral  $m_1$ . The sheets run first over the vertical discs *h* and  $h_1$ ,

and others) and are then taken out and dried, a firm coating of the dissolved material is formed over the piece. A dipping of about half a minute in duration is sufficient, for example, to provide a piece of gun-cotton with a coating half a millimetre thick." This coating serves as a protection against sealing or peeling, prevents or delays the penetration of moisture into dry pieces and delays the evaporation of moisture from wet pieces. "In addition to which the coating protects against the formation of fungus."—W. D. B.

## XXII.—ANALYTICAL CHEMISTRY.

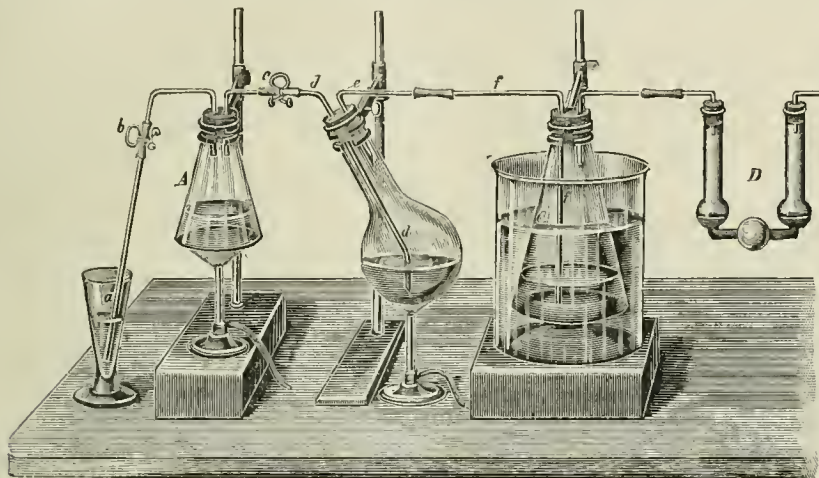
*A Simple Method for the Quantitative Estimation of Nitric Acid.*—Dr. E. Wildt and A. Scheibe. *Zeitschrift für Analytische Chemie.* 23, 151.

THE most accurate method of estimating nitric acid in presence of organic substances and ammonia is,

undoubtedly, that of Schloesing, in which the nitric acid is reduced to nitric oxide, the gas collected in a flask, reconverted into nitric acid by the introduction of oxygen, and the quantity determined by means of a titrated solution of caustic soda. The method requires very careful manipulation, and many attempts have been made to simplify it. In Tiemann's modification of Schulze's method, the nitric oxide is collected over caustic soda, and its volume measured. The gas is, however, slightly soluble in water, and may be partially oxidized by the oxygen dissolved in the water. Schloesing attempts to obviate this source of error as well as the calculations for temperature and pressure, by working always under the

the caustic soda in B, and the nitric oxide on coming in contact with the air in C is oxidised, and the nitric acid absorbed by the water. In case the current of gas is too rapid, the escaping nitric acid is absorbed in D. After an hour the tubes E and F are disconnected, while the solutions in A and B are still boiling, and the nitric acid is titrated with dilute caustic soda (about  $\frac{1}{4}$  normal). The vessel C must be well cooled during the whole experiment, which occupies about an hour and a half.

The results are accurate when the amount of nitric is not less than 0.25gm.; with smaller quantities they come out too low. Good results were obtained with potassium nitrate, Chili saltpetre, both alone and mixed with am-



same conditions, employing as nearly as possible the same quantity of nitric acid, and determining experimentally the amount of nitric acid corresponding to i.e. of NO. The objection still remains that other gases than nitric acid may be evolved, especially when the juices of plants are analysed. Boehmer absorbs the nitric oxide in a Liebig's potash apparatus containing chromic acid, and calculates the nitric acid from the increase in weight.

The author proposes the following modification of Schloesing's original method, with which he obtains very good results.

A is an Erlenmeyer's flask of 250c.c. capacity, containing the solution to be analysed. B is a round-bottomed flask of 250-300c.c. capacity, half filled with caustic soda, to absorb any hydrochloric acid which might be carried over from A. C is an Erlenmeyer's flask of 750c.c. capacity, containing a little water to absorb the nitric acid. D is a Peligot's tube, containing water to collect any nitric acid not absorbed by the water in C. The tube D is bent, as shewn in the diagram, and drawn out to a point, to diminish the size of the bubbles. The tube E is wide, and cut obliquely, to prevent water collecting and passing into C.

The experiment is conducted as follows: The clip B is closed and C opened, and the tube E disconnected from F. The solutions in A and B are then boiled for 20 minutes to remove all oxygen. The tubes E and F are again connected, the clip C is closed, the flame under B increased to prevent the liquid in C from being drawn back, and the clip B is opened. As soon as steam issues from the tube A, it is dipped into a conical glass containing 50c.c. of ferrous chloride prepared according to Schloesing's directions, and the flame under A is removed, when the ferrous chloride enters the flask. The clip B is regulated with the finger and thumb, so as to prevent the entry of air into the flask. The conical vessel is rinsed two or three times with water, and this is allowed to enter the flask, and the clip B is then closed, and the vessel A heated. The liquid in A turns brown in a short time, and nitric oxide is evolved. The clip C is opened slightly from time to time until the pressure is high enough, when it is opened entirely. The flames must be regulated so that a slow current of gas bubbles through the water in C. The hydrochloric acid is removed by

monium sulphate, superphosphate, and amido compounds. With superphosphate the solution should be made slightly alkaline, to prevent the liberation of nitric acid.—S. Y.

## New Books.

ON THE DISCOVERY OF THE PERIODIC LAW, AND ON RELATIONS AMONG THE ATOMIC WEIGHTS. By JOHN A. R. NEWLANDS, F.I.C., F.C.S. London: E. and F. N. Spon, 16, Charing Cross. New York: 35, Murray Street, 1884.

Svo volume, bound in cloth, contains 39 pages of matter.

The first few lines of the preface explain the intention and aim of the work, as follows:—

"This little book contains an exact reprint of all the papers on Relations among the Atomic Weights, and on the Periodic Law (provisionally termed the Law of Octaves), written by myself, and printed in the *Chemical News* some years before M. Mendelejeff had published anything on the subject of the periodic law." The appendix contains notes, etc., which have been published since the appearance of M. Mendelejeff's statements regarding the periodic law."

The quotations given on pages 37 and 38 (appendix) shew that, however tardily Newland's views were received at first, by recent writers of standard works on chemistry amongst his fellow-countrymen, his claims to priority are received.

A SHORT TEXT-BOOK OF INORGANIC CHEMISTRY. By Dr. HERMANN KOLBE, Professor of Chemistry in the University of Leipzig; translated and edited by T. S. HUMPHIDGE, Ph.D., B.Sc. (Lond.), Professor of Chemistry and Physics in the University College of Wales. With a coloured table of spectra, and numerous wood engravings. London: Longmans, Green and Co., 1884.

THIS work consists of an Svo volume, bound in cloth, and containing editor's and author's prefaces, a table of contents, 571 pages of subject matter, an appendix



containing 20 pages of matter, and an alphabetical index. With the text 66 woodcuts are interspersed. The work is of course a translation of the German textbook on Inorganic Chemistry, by Professor Kolbe, but certain alterations and additions have been made by the editor, with the author's consent, the chief additions occurring in the descriptions of water, air, coal, iron, etc. Short accounts of Gay Lussac's Law, Avogadro's Law, and the manufacture of coal gas have also been introduced, and the appendix already referred to contains further additions by Professor Hunnidge, in the shape of the methods used for determining atomic and molecular weights, of Prout's Law, and of the Periodic Law.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1884.

7958 A. R. Hancox, Haverhill, Suffolk. An improved liquid or compound for the removal of marks of grease and stains from woollen and other materials. May 20

7963 P. M. Justice, London. Improvements in treating or enriching phosphates of calcium or lime. Communicated by W. H. Keun, Luxembourg. May 20

7971 A. R. Leask, A. Thompson, and F. Ince, London. A new material to be used for the making of type for printing and other purposes. May 20

7976 W. H. Tooth, London. Improvements in the treatment of peat, and in the machinery or apparatus therefor, part of the apparatus being applicable to other purposes. May 20

7977 W. H. Tooth and J. E. Rooker, London. An improved method of and apparatus for treating lead, zinc, arsenic, sulphur, and other volatile metals, or the ores or minerals containing such volatile metals. May 20

7981 A. Schnell, Berne, Switzerland. Improvements in the treatment of malt and other grain. May 20

7982 C. D. Abel, London. A new or improved process for obtaining sugar from saccharine substances. Communicated by Dr. L. Harperath, Cologne, Germany. May 20

8020 A. F. Westerlund, Stockholm, Sweden. Improvements in producing cold, hard of combustion, of vegetable materials for hygienic and technical purposes. Complete specification. May 21

8021 A. D. Cohen and G. Nash, London. Perfumed antizymotic printing and writing inks, oil paints, and varnishes. May 21

8029 E. Du Bois Lukis, London. Improvements in the treatment of sulphate of iron or coppers for the production of pigments and sulphurous acid therefrom. May 21

8034 W. R. Lake, London. An improved method or apparatus of distillation, and apparatus therefor. Communicated by Wirth and Co., for Dr. F. Konig, Asti, Italy. May 21

8036 A. M. Clark, London. An improved process of hardening balsams, resins, and resinous compounds or products. Communicated by Wirth & Co. for A. K. Kissel and C. Zimmer, Frankfurt-on-Main, Germany. May 21

8037 A. M. Clark, London. The manufacture of a substitute for caoutchouc and gutta-percha. Communicated by Wirth & Co. for J. J. Harg and Caspar Hoffmann, St. Petersburg, Russia. May 22

8053 W. S. Sutherland, Birmingham. Improvements in and applicable to gas-producers. May 22

8056 A. Irving, Wokingham. Effecting the complete purification of water contaminated by dissolved vegetable matter. May 22

8083 S. J. Coxeter and H. Nehmer, London. The manufacture of silico-carbon. May 22

8093 G. Bischof, London. Improvements in the manufacture of spongy iron, and in apparatus employed therefor. May 22

8094 G. Bischof, London. Improved means and apparatus for the purification of water by spongy iron. May 22

8132 A. C. L. Weigel, Brighton. An improved manufacture of non-alcoholic beverages from grapes or other fruits. May 23

8135 T. Berliner, Ohlau, Germany. Process for cleaning bones, especially for glue manufacturing. May 23

8141 A. W. L. Reddie, London. Improvements in the setting of retorts and muffles. Communicated by the Stettiner Chamotte Fabriks Actien Gesellschaft, vormals Didier, Stettin, Germany. Complete specification. May 23

8148 T. Kempf, Ph.D., Berlin. Improvements in the process of manufacturing iodoform, bromoform, and chloroform. May 23

8166 T. Duncan and D. Mills, Heywood. A retort for brazing, melting metals, heating irons, etc., by combustion of gas and air. May 26

8171 J. W. Johnston, Sheffield. Improvements in tanks for cooling yeast. May 26

8186 J. Hargreaves, Widnes, and T. Robinson, Farnworth, Lancashire. Improvements in the construction and arrangement of apparatus or appliances for condensing hydrochloric acid gas. May 26

8200 J. Cornforth, Birmingham. Improvements in apparatus for consuming smoke and saving fuel. May 3 (previously included in No. 7169).

8210 M. P. W. Boulton, Tew Park, Oxfordshire. An improved method and apparatus for generating steam or vapour. May 26

8212 E. Ashby, Southampton, and A. Ashby, Grantham, Lincolnshire. Process and apparatus for decolorising the fumes of cement and other kilns. May 26

8218 T. Kempf, Ph.D., Berlin. Improvements in the process of manufacturing permanganates. May 26

8223 P. M. Justice, London. Improvements in refractory bricks and furnace linings. Communicated by T. Twynam, temporarily in North Africa. May 26

8234 J. Farmer, Salford, Lancashire. Improvements in the construction of apparatus for dyeing and washing fibrous materials. Partly communicated by A. Lalance, Mullhouse. May 27

8260 A. M. Clark, London. An improved method of and apparatus for treating saccharine juices. Communicated by L. M. Campi, Havana, Cuba. Complete specification. May 27

8263 E. Capitaine, London. Improvements in apparatus for fixing coatings of paste, pulp, and other thin substances. Communicated by W. Wepler, Offenbach-on-Main, Germany. May 27

8266 T. Higgin, Liverpool. Improvements in apparatus for disintegrating and removing impurities from lump salt. May 27

8268 R. Hadfield, Sheffield. Improvements in the treatment of steel. Complete specification. May 27

8281 P. M. Justice, London. Improvements in treating cotton seed. Communicated by the United States Cotton Seed Cleaning Co. New York. May 27

8285 G. F. Redfern, London. An improved apparatus for preparing oxygenated air. Communicated by A. Stamm, Leadville, U.S.A. Complete specification. May 27

8287 A. Marescot, London. Improvements in the method of and apparatus for the distillation of herbaceous substances. May 27

8295 J. A. Mays, Westminster. An improved method of amalgamating or separating metals from their ores. May 27

8296 F. M. Lyte, Putney, Surrey. The manufacture of a substitute for whalebone. May 27

8320 J. P. Jackson, Liverpool. Improvements in apparatus for filtering liquids. May 28

8332 J. Hantrive, Lille, France. A new or improved compound for protecting metals from corrosion or oxydation, and for other purposes. May 28

8340 E. G. Brewer, London. Improvements in the mode or process of and apparatus for concentrating, separating, or dividing mixtures of liquids. Communicated by P. de Puydt and H. Courtois, Brussels. May 28

8348 W. W. De La Rue, London. Improvements in the manufacture of paper and in apparatus employed therein. Complete specification. May 28

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The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

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 Wm. Dixon, jun., 2, Athol Road, Sunderland, works manager.  
 Augustus J. Farmer, 15, Park Place, Greenwich, S.E., tar distiller.  
 Ralph C. Forster, 19, Osborne Avenue, Newcastle-on-Tyne, merchant.  
 Duke Fox, Calder Bank Mills, Dewsbury, mungo and shoddy merchant.  
 Gorman Green, 2, Croft Terrace, Jarrow-on-Tyne Works, manager.  
 Fred. S. Hempleman, Wennington House, Wennington, Romford, Essex, manure manufacturer.  
 Deming Jarves, Michigan Carbon Works, Detroit, Michigan, U.S.A., manufacturing chemist.  
 George Johnson, Grange Metal Works, Jarrow-on-Tyne, copper manufacturer.  
 Walter Marsh, 4 York Terrace, Northam, Southampton, engineer.  
 Alf. Raphael, 87, Alexandra Road, London, N.W., merchant.  
 Dr. Adolf Spiegel, Hoechst am Main, Germany, chemist.  
 Francis J. Tennant, Belle Vue Terrace, Saltwell Lane, Gateshead-on-Tyne, chemical manufacturer.  
 John Wilton, Clydesdale, Norwich Road, Forest Gate, E., candle manufacturer and distiller.  
 Joshua Wright, 897, Ashton Old Road, Fairfield, near Manchester.

## CHANGES OF ADDRESS.

Robt. Fullarton (late of Carnoustie), 30, Donegall Place, Belfast, Ireland.  
 H. B. Fulton (late of Venezuela), c/o N. G. Burch, Esq., 1, Tokenhouse Buildings, London, E.C.  
 Ellwood Holmes (late of Manchester), Wellburn, Jesmond, Newcastle-on-Tyne.  
 F. G. Holmes (late of Peckham), Prince Regent's Wharf, Victoria Docks, E.  
 Alf. Geo. Jackson (late of Harpurhey), Oakleigh, Hale, Bowdon, Manchester.  
 Jno. Jas. Knight (late of Solihull), Hope Cottage, Shirley, Birmingham.  
 Thos. Macfarlane (late of Montreal), Uddingston, near Glasgow.  
 Jno. Rae (late of Oldbury), Quinton Villa, Quinton, near Birmingham.  
 Wm. Ray (late of Leeds), Bordesley Paper Works, Birmingham.  
 H. Robinson (late of Princess Street), 5, Cromford Court, Manchester.  
 Ferd. Rohling (late of Busby), Springbrook Works, Middleton Road, Oldham.  
 A. Schier (late of Bloomsbury Square), Thornfield, Harold Road, Upper Norwood, S.E.  
 Dr. J. H. Smith (late of Ponte Seveso), Wohlgelegen, bei Mannheim, Germany.  
 Alf. Wilson (late of Handsworth), gas furnace engineer, Stafford.  
 G. H. Bailey (late of Owens College), Barnard Castle, Co. Durham.  
 Adrian Blaikie (late of Manchester), 30, Osborne Road, Jesmond, Newcastle-on-Tyne.  
 J. H. Collins (late of Rio Tinto), 64, Bickerton Road, Highgate New Town, London, N.  
 E. C. Conrad (late of Bordeaux), 23, Well Street, Wellesloe Square, London, E.  
 Robert Williamson (late of Wilton Terrace), Osborne Villas, Cheetham Hill Road, Manchester.

## PROCEEDINGS OF THE THIRD ANNUAL MEETING,

*Held in the Amphitheatre of the Institute of the Literary and Philosophical Society of Newcastle-on-Tyne, Wednesday, July 9th, 1884.*

The PRESIDENT (Walter Weldon, Esq., F.R.S.) took the chair at eleven o'clock, and was supported by Dr. W. H. Perkin, Professor Roscoe, Mr. I. Lowthian Bell, and by representatives of the leading chemical firms throughout the United Kingdom. The Mayor of Newcastle (Dr. H. W. Newton) and the Sheriff (Mr. T. Nelson) attended the meeting in state, accompanied by the city mace and sword-bearers.

The PRESIDENT, in opening the proceedings, said they were honoured by the presence at their meeting of his Worship the Mayor of the city in which they were met, and also of the Sheriff of Newcastle, who were there for a most gracious purpose, and who were very short of time, as they were in attendance upon the Judges. He, therefore, proposed to ask them to be so good as to address the meeting before he called upon the General Secretary to read the minutes.

The MAYOR said his address to them would be extremely brief; but he was sure it would be none the less acceptable. His duty as chief magistrate of the city of Newcastle was to give the Society for the Promotion of Chemical Industry a most hearty welcome. It did appear that a society such as that could not possibly have selected a more suitable place for its annual gathering. Chemical industry, since the latter part of the last century, had been one of the staples of that district. It was not his province to enter into the technicalities of their work, because, with such names in the past as Losh, Doubleday, and Dundonald, and with such names in the present as Mr. Isaac Lowthian Bell, their worthy President, Mr. Swan of electric light celebrity, and a galaxy of others, he thought they had ample evidence that Newcastle had played a large part in the interest which they desired to further. Once more he gave them a most hearty welcome, and he did sincerely trust that their visit to Newcastle would be agreeable to themselves, and that it would leave behind some marks which would tend to promote the industry not only in that district, but over the whole country in the future.

The SHERIFF said that after the appropriate remarks of his colleague the Mayor, in bidding them welcome to this ancient city—or rather ancient town, for as a city Newcastle was only modern—he felt it would be unnecessary for him

to occupy their time. They were assembled there for a specific and important business, and he would not dilate upon the objects of their meeting. He thought he would suit their convenience by endorsing, as heartily as he possibly could, the reception that the Mayor had given them to that city.

The PRESIDENT said he was sure that they most profoundly appreciated the very great courtesy which had led to the presence with them that morning of his Worship the Mayor of that famous city, and its chief executive officer, the Sheriff, and which had dictated the most gracious words of welcome which had just been addressed to them. Some of them would not be so familiar with Newcastle as he had had occasion to become; but for his own part, he had long known that if there was anywhere a community which was always hospitable and gracious to a stranger within its gates, that community was the one which was presided over by his Worship the Mayor of Newcastle-upon-Tyne. And in the very hearty and gracious welcome which had been tendered to them that morning, and above all, in the seizing of the very earliest opportunity of offering that welcome, they had simply a representative instance, which he was sure they would all hold long in most pleasant remembrance, of a characteristic for which Newcastle was not less famous than for its High Level Bridge, its noble river, its countless coal pits, and the marvellous series of industrial establishments which rendered the spectacle which they would see as they went down the river to Hebburn and Tynemouth on Friday one of the most striking to be witnessed on the banks of any industrial river in the world. He begged again to thank the Mayor and the Sheriff sincerely for their most gracious welcome, and to invite them to do the Society the great honour of attending their dinner that evening. By their presence they would add very materially to the pleasure and satisfaction of all the members present at that, their first, but he trusted not their last, general meeting in that famous city.

Mr. CRESSWELL (the General Secretary), after reading the minutes of the last Annual Meeting held in London, which were confirmed, then read the

#### REPORT OF THE COUNCIL.

The Council has much satisfaction in reporting that during the last twelve months the Society of Chemical Industry has continued to make good progress. At the date of its first General Meeting, in July, 1881, the number of members on its register was 297. By July, 1882, this number had increased to 1,140, and by July last year to 1,390. Including 25 candidates elected this morning, the number of members of the Society is now 1,809. Moreover, 120 non-members now subscribe to the Society's Journal.

The Council has to deplore the loss during the past year of some of the most distinguished members of the Society, including Sir William Siemens, Mr. Peter Spence, Mr. Cromwell Varley, and Dr. Angus Smith. Sir William Siemens and Mr. Cromwell Varley, among the most illustrious workers of their generation in other fields, were not specially connected with the chemical industries; but Mr. Peter Spence was not only himself a chemical manufacturer, but was the author of many improvements in industrial chemical processes; and Dr. Angus Smith, besides being the author of a most valuable series of researches in pure chemistry, nearly always directed to the promotion of the health and well-

being of mankind, was intimately connected with the most important of the chemical industries as the first Chief Inspector under the Alkali Acts, in which capacity he performed the duties of a most onerous position in such manner as to render most important service alike to the public and to those engaged in the industries placed under his inspection.

During the past year two new local Sections of our Society have been established: one in Birmingham, with Mr. Alexander M. Chance as chairman, and one in Glasgow, with Mr. Edward C. C. Stanford as chairman. Six local Sections of the Society are thus now in operation, and the total number of papers which has been read before them during the session which has just closed is 68, divided as follows:—Birmingham Section 3, Glasgow Section 7, Liverpool Section 14, London Section 20, Manchester Section 17, and Newcastle Section 7. Many of these papers are of great practical value and utility, and to their authors, as well as to the executive officers of the various Sections, the best thanks of the Council and of the Society are due.

The question of an international arrangement with respect to methods of sampling and testing has been discussed by each of the local Sections of the Society, and a resolution on this subject will be submitted to this meeting.

Under the editorship of Mr. Watson Smith, and the supervision of the Publication Committee, the Society's Journal, during the past year, has at least maintained its former position, and its value to all who are engaged in the practice of applied chemistry is now widely recognised. The increase in the number of our Local Sections, however, has necessarily involved an increase in the size of the Journal, and therefore in its cost; and to meet that increased cost a still further increase in the number of our members is essential.

In order to avoid controversy in the columns of the Journal, it has been decided to cease to admit into it "communications" which have not been read before the Society or one of its Sections.

In response to many applications from new members for copies of the first volume of the Journal, the stock of certain numbers of which had become exhausted, the Council determined some time ago to reprint the numbers in question, and copies of the first volume will very soon be obtainable.

Experience has shown that some slight modifications of the Society's Bye-Laws are essential; and the modifications which the Council, after careful consideration, has decided to propose, and which the members will be asked to accept at this meeting, were published in the May number of the Journal. If accepted by the members, these modifications will widen somewhat the "Objects" of the Society, as defined by its Bye-Laws as these at present stand, though it is believed not beyond the intention of the founders of the Society; will indicate the course to be taken when unexpected vacancies occur in the Council and the local Sections of the Society; will render retiring Presidents eligible for re-election; but will otherwise make changes only of wording and of arrangement.

On the motion of the PRESIDENT, seconded by Mr. NEWALL, the Report of the Council was adopted.

The SECRETARY then read the Treasurer's report.



## SOCIETY OF CHEMICAL INDUSTRY.—TREASURER'S STATEMENT, DECEMBER 31st, 1883.

			£	s.	d.				£	s.	d.
To Cash on Deposit (1st January, 1883) .....						300	0	0	By Journal Expenses:—		
Balance at Bank .....						£372	2	3	Editorial .....		
Ditto in hand .....						26	5	0	Publishing .....		
						£398	7	3	1,187 6 10		
Less amount owing to Hon. Secretary .....						4	0	10			
						394	6	5			
Annual Subscriptions:—											
180 for the year 1882 .....						£189	0	0			
1,316 for the year 1883 .....						1,381	16	0			
104 for the year 1884 .....						109	4	0			
50 for the year 1885 .....						52	10	0	100 8 4		
						1,732	10	0			
Entrance Fees .....						113	8	0			
Life Composition Fees .....						50	0	0	108 6 8		
Interest on Deposit Account .....						16	7	3	122 2 6		
Macmillan and Co. (publishing "Proceedings") .....						2	0	6	43 17 9		
Journal:—									9 14 1		
Advertisements .....						£99	0	0			
Sales of .....						£118	10	9			
Less Publishers' Com- mission .....						28	16	8			
						89	14	1	8 15 9		
						188	14	1	56 10 0		
									50 0 0		
									5 5 0		
									126 0 0		
									0 19 7		
									2 10 6		
									27 18 2		
									11 0 8		
									936 19 7		
									£2,797 6 3		

			£	s.	d.				£	s.	d.
Cash on Deposit, 31st December, 1883 .....						£500	0	0			
Balance at Bank .....						423	13	3			
Do. in Treasurer's hands .....						12	12	0			
Do. in Secretary's hands .....						0	11	4			
						936	19	7	£2,797 6 3		

Audited and found correct this 13th day of March, 1884.

DAVID HOWARD,  
RUDOLPH MESSEL, } AUDITORS.

## SUPPLEMENTARY STATEMENT, JUNE 30th, 1884.

		Dr.	£	s.	d.	£	s.	d.	Cr.	£	s.	d.	£	s.	d.		
To Cash on Deposit (31st Dec., 1883).....						500	0	0	By Journal Expenses:—								
Balance at Bank „ „ .....						423	13	3	Editorial.....						240	11	8
Do. in Treasurer's hands „ .....						12	12	0	Publishing.....						640	2	2
Do. in Secretary's hands „ .....						0	14	4									
Annual Subscriptions:—									General Printing .....						880	13	10
5 Subscriptions for the year 1882 .....						5	5	0	Sectional Expenses:—								
85 „ „ „ 1883 .....						89	5	0	Birmingham Section .....						11	0	2
1428 „ „ „ 1884 .....						1499	8	0	Glasgow „ .....						54	6	8
6 „ „ „ 1885 .....						6	6	0	Liverpool „ .....						27	5	9
1 „ „ „ 1886 .....						1	1	0	London „ .....						52	8	1
Amounts overpaid (less Bank									Manchester „ .....						57	13	8
Charges on Scotch and Irish									Newcastle „ .....						23	7	0
Cheques) .....						0	0	4									
Life Composition Fees .....						60	0	0	Secretary's Salary .....						226	1	4
Interest on Deposit Account .....						9	7	11	Amount voted by Council to Secretary (for						100	0	0
Journal:—									extra work in 1883) .....						25	0	0
Advertisements .....						34	10	0	Amount voted to E. Hughes, by Council (for						21	0	0
Sales .....						£103	1	4	assistance rendered to Hon. Treasurer) ....						20	19	9
Less Commission to									Stationery .....						8	10	6
Publishers .....						£15	15	1	Office Furniture.....								
						87	6	3	Office Expenses:—								
									Rent .....						33	15	0
									Sundries.....						8	5	6
</																	

On the motion of the PRESIDENT, seconded by Mr. A. M. CHANCE, the Report of the Honorary Treasurer was adopted.

The PRESIDENT, who, on rising, was received with loud applause, then delivered the following address:—

THE ADDRESS OF THE PRESIDENT.

I trust that it will not be unfitting that the observations which it is now my duty to offer should be devoted exclusively to that one of the chemical industries of which the district in which we are now met is so important a centre, which was first practised in England within four miles of this building, and with which it happens that I am more familiar than with any other. Eighteen months ago, before the London Section of our Society, I sketched the then present condition of that industry, and indulged in some speculations as to its future. "Many things," however, "have happened since then;" and among them have been changes, alike in the actual condition of the older branch of the Soda Industry, and in the data for estimating the probable future of that industry at large.

Since I collected, towards the end of 1882, the statistics with respect to the Soda Industry which I presented to our London Section in January last year, the ammonia process has continued its victorious advance, and the production of Leblanc soda has still further diminished. That further diminution in the production of Leblanc soda was indeed inevitable; but, whereas one expected that it would involve the closing of more Leblanc soda works: temporarily, at any rate, this result has been avoided by combinations among Leblanc soda makers to reduce production. These combinations have been regarded as simply combinations to force up the price of certain products; but there is no room to doubt that the rise which has taken place in the price of the products in question, as a consequence of these combinations, must necessarily have taken place by this time, if those combinations had not been formed. The manufacture of chlorine and Leblanc soda, without profit, and in many cases at a loss, could not have gone on much longer. It must soon have resulted in the forced withdrawal of the weaker manufacturers from a hopeless struggle; and the effect upon prices of diminished production brought about in this way would of course have been the same as that of diminished production brought about by mutual agreement. The stronger of the manufacturers might well have preferred a continuance of the struggle until all the weaker had gone to the wall; but the consumer has certainly no cause to complain of an arrangement: the faint flavour of socialism about which renders it none the less acceptable in at least my eyes: by which,—instead of the complete closing of more works, and the ruin of their owners, the other works remaining in full activity and reaping the whole benefit of the reduced production thus occasioned,—on the one hand, the reduced production which must have been brought about in some way, and, on the other, the benefit of the higher prices resulting from it, are shared, as equally as may be, among the proprietors of all the works which were still in operation when that arrangement was made.

While, however, the makers of soda by the Leblanc process are to-day in the position, to which they had so long been unaccustomed, of being able, not only to make both ends meet, but also, I trust, to make one end somewhat overlap the other,—what of the future of that process? Regarded merely as a means of manufacturing soda, it has served its time. Per given quantity of soda yielded by it, it is more than seventy per cent. more costly than the ammonia process; and thus its only claim to continue to live consists in the fact that it yields hydrochloric acid. So far as one

can yet see, the world must continue to be supplied with chlorine. At present, the immediate raw material of the chlorine manufacture is always hydrochloric acid; and for our supply of that raw material we are entirely dependent on the Leblanc process. Is this state of things destined to continue? Is the Leblanc process likely to remain our only means of obtaining hydrochloric acid? And, above all, must the production of hydrochloric acid always be, as now, an essential condition precedent of the production of chlorine?

How to obtain in a useful state the chlorine, at present thrown away as calcium chloride, of the salt which is decomposed by the ammonia process, is a problem which has long occupied many minds. When it became known, in the course of last year, that Mr. Mond had patented a process to that end, it was widely felt that the solution of this problem had probably been reached; and Mr. Mond's very ingenious process certainly would, under given conditions, enable the manufacturer of ammonia soda to utilise both the constituents of the salt which he decomposes. Mr. Mond himself, however, has never regarded this process as other than a process for the future. It is a process which would involve that the ammonia employed in the manufacture of ammonia soda, instead of being continually regenerated and used again as at present, should be used as a reagent in that manufacture only once, and should then be sold as ammonium sulphate; and hence the conditions for its extensive application obviously do not at present exist.

The form of Mr. Mond's process is due to a fact of which, until he discovered it, chemists were not aware. We know that when sodium chloride is heated with an equivalent of sulphuric acid, as in the first operation of the Leblanc process, the sulphuric acid reacts on only one-half of the sodium chloride, the products of its reaction thereupon being HCl and hydrogen sodium sulphate; but that afterwards at a higher temperature, that hydrogen sodium sulphate reacts on the other half of the sodium chloride, another HCl being evolved, and the whole of the sodium present being converted into normal sulphate. It has been generally supposed that if ammonium chloride were substituted for sodium chloride, things would go in a similar way; but Mr. Mond has found that this is not the case. He has found that when ammonium chloride and acid ammonium sulphate are heated together, no reaction takes place between them, at whatever temperature. Below a certain temperature, nothing happens; above that temperature, all that happens is that ammonium chloride volatilizes. That this fact remained to be discovered in 1883, surely shews that, in the search for new organic compounds in which chemists for so many years past have been so absorbed, the pursuit of inorganic chemistry has been somewhat unduly neglected.

When the residual liquor of the ammonia soda process has been treated for the distillation off from it of such part of its ammonia as is combined with carbonic acid, together with such of it, if any, as may be in the free state, what remains is a nearly saturated mixed solution of ammonium and sodium chlorides. On evaporating this mixed solution, its sodium chloride, being the less soluble of the two salts contained in it, falls, and may be fished out; leaving eventually a mother-liquor consisting of solution of ammonium chloride only. Mr. Mond's proposed process consists in separating in this way the sodium chloride of the mixed solution in question; in then evaporating the mother-liquor to dryness, so as to obtain solid ammonium chloride, and in then treating this solid ammonium chloride by enough sulphuric acid to form acid ammonium sulphate,  $\text{H}_2\text{NH}_4\text{SO}_4$ , the



chlorine of the ammonium chloride going off as HCl. The residual acid ammonium sulphate is then either to be treated by vapour of free ammonia, and so converted into normal ammonium sulphate, for sale as such, or it is to be used, instead of free sulphuric acid and normal ammonium sulphate, in the manufacture of richly ammoniacal phosphatic manures.

If one wished to convey an idea of the stupendous scale upon which ammonia soda is now manufactured, I think one could not do it better than by pointing out some of the conditions which would be necessary to the application of this process to the whole of the ammonium chloride produced by a single ammonia soda maker, Mr. Mond himself. It is no secret that Mr. Mond manufactures fully 50,000 tons of ammonia soda per annum. To treat the whole of the ammonium chloride corresponding to this quantity of ammonia soda by that form of the process in question in which the acid ammonium sulphate at first produced should be afterwards converted into normal ammonium sulphate by means of free ammonia, would require, not only that he should manufacture per annum 128,000 tons of ammonium sulphate, but also that he should command a constant supply of one-third more ammonia than the total quantity at present produced in all Great Britain and Ireland; while to treat that vast quantity of ammonium chloride by the form of his process in which the acid ammonium sulphate intermediately produced should be employed to react on calcium phosphate, he must manufacture per annum a quantity of manures containing 64,000 tons of ammonium sulphate, which quantity of manures could not, I believe, be less than 350,000 tons, or 40 tons for every hour in the whole year, and the production of which would require a supply of ammonia equal to two-thirds of the total quantity at present produced in all the gas-works of the United Kingdom. It would involve the annual consumption of a quantity of ammonia, the production of which in gas-works at present requires the distillation of five millions of tons of coal, and the concentration of which, as gas-liquor, upon any one spot, would require that gas-liquor should come pouring in there at the rate of from a ton to a ton-and-a-half per minute, night and day, week-days and Sundays, all the year through.

Obviously,—and, as I have said, I know that this is Mr. Mond's own view,—this process cannot be extensively applied so long as we continue dependent for our supply of ammonia mainly on the distillation of coal for the manufacture of illuminating gas; and cannot, indeed, be applied on any serious scale at all except in proportion as means are realized of obtaining as ammonia the nitrogen of coal used as fuel, and as those means are practised in the immediate neighbourhood of ammonia soda works. Such means doubtless will be realized; but very much will happen before the supply of ammonia becomes so abundant as to permit the ammonia soda maker to use his principal reagent only once and then to sell it; and when that time at length arrives I think he will have no temptation to treat his ammonium chloride by sulphuric acid, but if he desires to sell his ammonia, after having used it only once, will probably best find his account in selling it in the form in which his soda process yields it. Meanwhile, I believe that Mr. Mond is not only not preparing to put this process into operation, on any scale whatever, but has no immediate intention of doing so.

An idea for the obtaining of hydrochloric acid in connection with the ammonia process, which has found more favour in many eyes than I think it deserves, and has recently been the subject of very much experiment, is one involving a modification of the ammonia process itself, by the substitution of sodium sulphate for sodium chloride as its starting

point. The idea is to first decompose salt by sulphuric acid, and so obtain hydrochloric acid and sodium sulphate, as in the Leblanc process at present, and then to treat that sodium sulphate by ammonia and carbonic acid. The experiments which have been made in this direction at least place beyond doubt what before was widely disbelieved, namely, that a sulphate of soda ammonia process, whatever its commercial merits or otherwise, is in itself perfectly practicable. The lower solubility, in water at ordinary temperatures, of sodium sulphate than of sodium chloride, has been supposed to be an obstacle to the realization of such a process; but there is no inconvenience in employing, in the operation of decomposing sodium sulphate by bicarbonate of ammonia, a temperature of about 34° C., and at that temperature sodium sulphate is more soluble than sodium chloride, so that it is quite practicable to obtain, by treating by CO<sub>2</sub> an ammoniacal solution of sodium sulphate, more bicarbonate of soda than can be obtained by the corresponding treatment of an equal volume of ammoniacal brine. It has also been supposed that only one-half of the sodium sulphate treated by ammonia and carbonic acid would undergo decomposition, the second half of it going to form, with the ammonium sulphate resulting from the reaction of bicarbonate of ammonia on the first half of it, double sulphate of sodium and ammonium; but experience has proved that this is not so. Nor does the regeneration of ammonia by means of lime from the ammonium sulphate of the mother-liquor of a sulphate of soda ammonia process present any difficulty. The operation cannot be performed, owing to the formation in it of solid and bulky calcium sulphate, in precisely the same manner as the corresponding operation of the present form of the ammonia process; but means have been found of effecting it with an ease and completeness which leave nothing to be desired. It has been found, too, that that calcium sulphate can be readily separated from so much of the residual solution, containing a good deal of sulphate of soda, in which it is at first suspended; that,—the separated solution being used to dissolve the next quantity of solid sodium sulphate entering the process,—the loss of sodium sulphate in a sulphate of soda ammonia process need be only extremely small. It seems quite clear, therefore, that such a process would present no practical difficulty whatever.

But *cui bono*? To merely substitute for the Leblanc method of decomposing sodium sulphate its decomposition by bicarbonate of ammonia, would surely be a game not worth the candle. In the Leblanc process, as in so many other things, *c'est le premier pas qui coûte*. It is the first operation of the Leblanc process which makes that process so costly. It is the cost of his sodium sulphate which places the maker of Leblanc soda at such a disadvantage as compared with the maker of ammonia soda. If sodium sulphate cost the former no more than salt costs the latter, it is Leblanc soda, and not ammonia soda, which would be the cheaper of the two, since the cost of manufacturing Leblanc soda from sodium sulphate is appreciably less than the cost of manufacturing ammonia soda from salt. The treatment of sodium sulphate by ammonia and carbonic acid would at least not be less costly than the treatment of sodium chloride by the present form of the ammonia process; and hence to merely substitute for the Leblanc method of decomposing sodium sulphate the method of decomposing it by bicarbonate of ammonia would be to substitute for the later operations of the Leblanc process a series of more costly operations, having no advantage beyond one of scarcely appreciable commercial value: that of yielding a more concentrated product.

While, however, the only advantage of a process which should consist in manufacturing sodium sulphate as in the Leblanc process at present, and then decomposing that sodium sulphate by bicarbonate of ammonia, would thus be one which would not be worth the cost of obtaining it: a process, based on the decomposition of sodium sulphate by bicarbonate of ammonia, has recently been devised, which stands upon a very different footing. In the process to which I now refer, the sodium sulphate to be afterwards decomposed by bicarbonate of ammonia would be obtained, and hydrochloric acid at the same time, without consumption of sulphuric acid, except to make up mechanical loss, by means of one and the same quantity of sulphuric acid continually regenerated and used again.

This remarkable process is the invention of three members of the firm of Gaskell, Deacon & Co., of Widnes: Mr. Carey, Mr. Holbrook Gaskell, junr., and Dr. Hurter. These gentlemen have found a means of so decomposing ammonium sulphate as to obtain not only its ammonia in the free state, but also its sulphuric acid, if not as free sulphuric acid, at any rate in a state in which it can decompose sodium chloride.

Chemists have long known that when ammonium sulphate is heated to a certain temperature one-half of its ammonia is set free and flies off, the other half of it remaining behind as hydrogen ammonium sulphate. Messrs. Carey, Gaskell and Hurter have discovered that if a mixture of equivalent quantities of ammonium sulphate and sodium sulphate be heated to fusion, and steam be injected into the liquid mass, the whole of the ammonia of the ammonium sulphate is disengaged in the free state: its sulphuric acid combining with the sodium sulphate to form hydrogen sodium sulphate, which alone remains behind.

Upon this most interesting reaction there may obviously be based a soda process which should begin by obtaining sodium sulphate and hydrochloric acid by the reaction of "bisulphate of soda" on sodium chloride, the normal sodium sulphate so obtained being then decomposed by bicarbonate of ammonia, with production of bicarbonate of soda and ammonium sulphate, such ammonium sulphate being then so treated as to regenerate, by one and the same operation, both ammonia for use again, and bisulphate of soda for use again.

The question of whether or not this most original and beautiful process can be reduced to practice must obviously depend mainly, if not solely, upon the degree of readiness with which the new reaction upon which it is based can be carried to such completeness as to avoid any serious loss of ammonia. I am unacquainted with the experimental results on this point which have so far been obtained; but it is easy to see that that reaction might be somewhat slow, and, therefore, somewhat costly, and yet the process based upon it be one of very serious importance.

Charging against the hydrochloric acid yielded by Messrs. Carey, Gaskell and Hurter's process, the cost of those operations of their process which are in excess of the operations which the ammonia soda maker performs at present, one may count that the cost of their soda would be practically the same as that of ammonia soda as at present manufactured from brine. Their process would start from solid salt, which is, of course, more costly than brine; but, in some localities at any rate, the value of the lime which is required for the decomposition of the ammonium chloride of the present form of the ammonia process, but which would not be required in Messrs. Carey, Gaskell and Hurter's process, would fully compensate the greater cost for raw materials of the latter than of the former.

The operations the cost of which would thus have

to be charged against Messrs. Carey, Gaskell and Hurter's hydrochloric acid are those: firstly, of evaporating to dryness the mixed solution of ammonium sulphate and sodium sulphate constituting the mother-liquor of the bicarbonate of soda obtained by treating an ammoniacal solution of sodium sulphate by carbonic acid; secondly, of treating the residue of that evaporation, after addition to it of more sodium sulphate, for the regeneration of ammonia and the production of bisulphate of soda; and, thirdly, of then furnacing that bisulphate of soda with an equivalent quantity of salt.

The quantity of water which would have to be evaporated, in the first of these three operations, per ton of sodium carbonate manufactured, may be put, I believe, at five-and-a-half tons. It would be the water of a mixed solution of ammonium sulphate and sodium sulphate, and as these are both salts which, like sodium chloride, absorb heat during solution in water, and so give out heat during the inverse operation,—the two together, in the proportions in which they would be present in the solution in question, to just about the same extent as sodium chloride,—it ought to be possible to evaporate those five-and-a-half tons of water by means of the one ton of fuel which suffices for the evaporation of more than that quantity of water from brine. For the third operation, which would correspond to the second half of the first operation of the Leblanc process, less than half-a-ton of fuel should be ample. The labour upon these two operations would obviously be but slight, so that the cost of both operations together, for fuel and labour, per ton of sodium carbonate manufactured, could scarcely, I think, exceed eleven or twelve shillings.

The quantity of hydrochloric acid which corresponds to the quantity of sodium chloride corresponding to a ton of sodium carbonate, counted as aqueous acid of 27 per cent., is 2·548 tons. There are some Leblanc soda works in which the proportion of the total HCl evolved therein, which is obtained as aqueous acid of useful strength, is fully 95 per cent. Assuming this proportion of the HCl evolved in Messrs. Carey, Gaskell and Hurter's process to be obtained in an useful form, the process would yield, per ton of sodium carbonate manufactured by it, a quantity of aqueous acid corresponding to 2·42 tons at 27 per cent.

The value of this quantity of hydrochloric acid I estimate at £2 8s., and I arrive at that value for it in this way. The difference between the cost of ammonia soda and that of Leblanc soda *plus* hydrochloric acid, per quantity of Leblanc soda equivalent to a ton of ammonia soda, now averages, I believe, in England, about £2 15s. These £2 15s. I take to be the value, in the sense of the actual cost, not of the quantity of hydrochloric acid, of useful strength, which is usually obtained concurrently with that quantity of Leblanc soda, but, what is by no means the same thing, of the greatest quantity of such acid which is obtained, per such quantity of soda, in any instance within my knowledge. This quantity—for we must remember that the Leblanc soda maker has to produce more sodium sulphate, and, consequently, more hydrochloric acid, than merely the quantity equivalent to the alkali he finally obtains—is 95 per cent. of the quantity corresponding to about twelve-and-a-half per cent. more sodium sulphate than the quantity of sodium sulphate equivalent to a ton of sodium carbonate, and is thus, counted as acid of 27 per cent., almost exactly two-and-three-quarter tons: £2 15s. for which quantity is just £1 per ton, or £2 8s. for the 2·4 tons which I have taken to be obtainable, by the process under consideration, per ton of sodium carbonate manufactured thereby.

Taking it, then, that the minimum present cost of



aqueous hydrochloric acid of 27 per cent. is £1 per ton; that Messrs. Carey, Gaskell and Hurter's process could yield, per ton of sodium carbonate manufactured by it, 2·4 tons of such acid, thus worth at least £2 8s.; and that, of the three operations the cost of which would have to be charged against those 2·4 tons of hydrochloric acid, twelve shillings would pay for two: there would thus remain £1 16s., the balance left after the deduction from which sum of the cost of effecting Messrs. Carey, Gaskell and Hurter's reaction on about 2 tons 12 cwt. of a mixture of ammonium sulphate and sodium sulphate, would be the measure of the advantage of their process over the Leblanc process.

It is so difficult to conceive that the cost of effecting the reaction in question upon the quantity of materials which I have mentioned could be anything like £1 16s., that for this process of Messrs. Carey, Gaskell and Hurter's I could not but anticipate a most important future: were it not for the results of a renewed attempt which has for some time past been in progress to realize the old idea of decomposing the ammonium chloride of the present form of the ammonia process by magnesia, and then so treating the resulting magnesium chloride as to obtain its chlorine in a useful form and at the same time to regenerate magnesia for use again. In this method of procedure there would be one operation less than in Messrs. Carey, Gaskell and Hurter's process; the operation of evaporation, which would be practically the same in both processes, being followed, in Messrs. Carey, Gaskell and Hurter's process, by two operations, in the first of which there would be treated, per ton of sodium carbonate manufactured, about 52 cwt. of mixture of sodium sulphate and ammonium sulphate, and in the second about 68 cwt. of mixture of sodium chloride and hydrogen sodium sulphate, while, in what may be called the magnesia process, the operation of evaporation would have to be followed by simply the one operation of decomposing about eighteen-and-a-half hundredweight of magnesium chloride. To which must be added the far graver consideration that while Messrs. Carey, Gaskell and Hurter's process can yield the chlorine of the salt treated by it only as hydrochloric acid, a large part of the chlorine of magnesium chloride can be obtained directly in the free state.

It is MM. Pechiney et Cie., of Salindres, in the south of France, who have had the boldness to attack again the problem,—upon which so many earnest workers had spent in vain their utmost efforts during many years,—of how to obtain, as magnesium chloride, the chlorine of the salt decomposed by the ammonia process; and their genius and skill have enabled them to reach results which compel the conviction that the industrial realization of the decomposition by magnesia of the ammonium chloride of the ammonia process is now only a question of time.

If, then, the manufacturer of ammonia soda shall become able, as he certainly will become able, to obtain as magnesium chloride the chlorine which he at present throws away as calcium chloride: how is that magnesium chloride to be dealt with? When an aqueous solution of magnesium chloride is subjected to evaporation, vapour of hydrochloric acid begins to be disengaged so soon as the quantity of water present has fallen to six equivalents per equivalent of magnesium chloride. Fifteen or sixteen years ago, at the Gerard's Bridge Works of Messrs. J. C. Gamble & Son, of St. Helens, very considerable quantities of solution of magnesium chloride, concentrated to that point, were treated in muffle furnaces. The magnesium chloride was readily enough decomposed, but the furnaces were rapidly destroyed: by reason of the concentrated solution fed into them penetrating their beds, and causing the beds to "rise." The Gerard's

Bridge experiments on the decomposition of magnesium chloride were therefore abandoned; but not until they had proved that the difficulties in the way of decomposing that compound industrially by heating it in an atmosphere of vapour of water were simply mechanical difficulties.

Seeking, during many years subsequently, means of treating by heat and air, under industrial conditions, chlorides of various metals, I was at length led, in 1881, to the idea of adding to a concentrated solution of a chloride or mixture of chlorides, oxide of the same metal or metals, and then treating by heat and air solid masses of the resulting mixture; and since early in 1882 MM. Pechiney et Cie. have been earnestly engaged in endeavouring to realise this method of working, especially as applied to magnesium chloride, enormous quantities of which they obtain as a bye-product of the manufacture of salt from sea-water, and also to manganese chloride. In the case of magnesium chloride, the product of the addition to its aqueous solution of magnesium oxide is, of course, not a mere mixture, but the compound, magnesium oxychloride. I had found in the laboratory that this body, heated in contact with air under certain conditions, gives off an appreciable part of its chlorine in the free state; and the larger experiments which have since been made at Salindres leave no doubt that fully half the chlorine of magnesium chloride may be obtained directly as free chlorine by converting the magnesium chloride into oxychloride, and then heating that oxychloride, under certain conditions, in presence of air. What this means is that a manufacturer of ammonia soda, able to decompose his ammonium chloride by magnesia, will be able, by evaporating, per ton of ammonia soda manufactured, a quantity of solution of magnesium chloride containing about five and-a-half tons of water, and then heating in presence of air a mixture of about eighteen-and-a-half hundredweight of magnesium chloride with about eight hundredweight of magnesia, to obtain the quantity of free chlorine corresponding to very nearly a ton of bleaching-powder,—being the quantity of chlorine the present cost of which, as I shall show in a moment, is close upon £6,—and, in addition, a quantity of hydrochloric acid corresponding to something over a ton of aqueous acid of 27 per cent. The chlorine obtained by heating magnesium chloride in contact with air is of course diluted by other gases; but MM. Pechiney et Cie. have in operation an extremely simple mechanical bleaching-powder chamber, the complete absorption in which of the chlorine of a mixture of chlorine and other gases presents no difficulty.

The two questions which I asked some time ago I have now answered. Clearly, the Leblanc process is not destined to remain our only means of obtaining hydrochloric acid; and for my own part I can have no doubt that the use of hydrochloric acid as the immediate raw-material of the chlorine manufacture is also one of those things in respect of which the old order must,—partly, at any rate,—give place to new.

And I ought to add that the processes for the obtainment of chlorine and hydrochloric acid in connection with the manufacture of ammonia soda which I have mentioned, are not the only processes to those ends which are in the field. Within the last few weeks M. Solvay has taken three more patents for such processes; and quite a number of other workers at the problem of obtaining chlorine or hydrochloric acid from calcium chloride now consider that they are on the verge of success.

While, however, it is now quite certain that chlorine will be manufactured at least from magnesium chloride, one can hardly conceive that its manufacture

from either magnesium chloride or calcium chloride can ever more than partially displace its manufacture from hydrochloric acid, whatever changes may take place in our mode of obtaining hydrochloric acid. I come now, therefore, to the question of the utilization of hydrochloric acid in the chlorine manufacture more completely than it is utilised therein at present: a question upon which the continued existence of the Leblanc process certainly now mainly depends.

The present Weldon process, even when performed to the very best advantage, does not yield more than one-third of the total chlorine of the hydrochloric acid treated by it: the other two-thirds of that chlorine being lost as calcium chloride. It is thus unquestionably a barbarous process. It met well enough the wants of the time when hydrochloric acid could be counted as having no value, and when the two problems in respect of the chlorine manufacture which pressed to be dealt with were simply those of how to diminish the then chief item of the cost of chlorine,—that for native manganese,—and how to keep the offensive residual product of the treatment of that native manganese by hydrochloric acid out of the water-courses; but it is no longer equal to the production of cheap bleaching-powder, now that the value of hydrochloric acid has become such that the bleaching-powder produced by it costs more for hydrochloric acid than for all other items of its cost put together. For some years past, therefore, many inventors have been endeavouring to devise a hydrochloric acid chlorine process which should yield in the free state practically the whole of the chlorine of the acid treated by it; and a process which I believe will do this is one of the group of processes to the endeavour to realise which M.M. Pechiney et Cie. have for some time past been devoting not only vast material resources, but a power of overcoming the practical difficulties which are always encountered in attempts to work out new industrial chemical processes, of which I have known no parallel.

The process in question is simply a new form of a very old idea. Its first operation consists in digesting with aqueous hydrochloric acid an oxide of manganese which has been regenerated from manganese chloride in the dry way. There are thus obtained undiluted chlorine, and a residual solution of manganese chloride. The latter is then evaporated to dryness, and the solid manganese chloride so obtained is then heated in intimate contact with atmospheric oxygen. The chlorine of the manganese chloride is thereby driven off in the free state,—mixed, of course, with other gases,—and its manganese obtained as an oxide, rich in oxygen, with which to recommence the round of operations. Per given quantity of hydrochloric acid treated by it, the process yields practically the same quantity of strong chlorine as can be obtained from the same quantity of acid by means of Weldon mud, together with something over twice that quantity of chlorine diluted by other gases.

I shall certainly at least not be above the mark if I take the consumption of hydrochloric acid, per ton of bleaching-powder made by the present Weldon process, at the quantity the production of which by the Leblanc process, together with the quantity of soda which must be produced at the same time, costs £5 more than the cost of producing by the ammonia process the equivalent of that quantity of Leblanc soda: or, in other words, at the quantity corresponding to five tons of aqueous hydrochloric acid of 27 per cent. I know of no instance in England in which a ton of Weldon bleach is obtained per less than 5·7 tons of such acid; but I propose to assume that a ton of bleaching-powder can be made, by the present

Weldon process, per the quantity of acid the present minimum cost of which is £5.

The other items of the cost of a ton of bleaching-powder, made by that process,—apart from lime for the chambers, labour on the chambers, casks, packing, and general charges,—are those for lime for the oxidizers, limestone or chalk for the wells, manganese to make up loss, fuel, labour on the stills, and labour on the regeneration process. I believe that the sum of these items averages about 19s.: bringing up to about £5 19s. that part of the expense of manufacturing a ton of bleaching-powder by the present Weldon process which represents the cost of that ton of bleaching-powder for chlorine only.

The process which is now on the point of being got to work at Salindres, after much experience of it on a semi-industrial scale, requires neither lime nor limestone, nor does one see how there can be any appreciable loss of the manganese employed in it. The loss of manganese incurred in the present Weldon process is mainly due to impurities which are constantly entering that process, and have to be continually eliminated and thrown away, and with which it is impossible to avoid throwing away more or less manganese; but in the new process in question there is nothing to throw away. Its cost is thus simply for acid, fuel, and labour.

That that process is capable of yielding the quantity of chlorine corresponding to a ton of bleaching-powder, per quantity of hydrochloric acid corresponding to less than 30 cwt. of acid of 27 per cent., is already sufficiently demonstrated. The value of that quantity of acid being taken at £1 10s., there remains £5 19s., less £1 10s., or £4 9s., out of which to pay simply the cost of evaporating to dryness a quantity of solution of manganese chloride containing one-and-three-quarter tons of water, and then treating by heat and air something less than fifteen hundred-weight of manganese chloride mixed with a certain proportion of manganese oxide. The advantage of the new process over the old one, per ton of bleaching-powder manufactured, will be the balance left after deducting from those £4 9s. the cost of these two operations.

The difficulties which have been encountered in the endeavour to realize this process have been mainly with respect to the apparatus and mode of working by which to effect the often proposed reaction of atmospheric oxygen upon manganese chloride. Manganese chloride is a somewhat readily fusible salt; oxygen does react upon it below its fusing point, but only slowly; for the reaction to go on sufficiently rapidly, the temperature employed must be above the fusing point of the manganese chloride, and the latter must present to the air by which it is treated a very considerable surface; while a fused mass of manganese chloride not only presents far too small a surface, but is destructive of all materials of which apparatus for treating it industrially could be formed.

For the treatment of manganese chloride, as of magnesium chloride, the principle adopted at Salindres is that, to which I have already referred, of mixing the chloride to be treated by heat and air with an oxide of the same metal: the main function of the oxide being to mechanically divide the chloride, so as to permit of the chloride being heated to above its fusing point, without the mass treated becoming fluid, or ceasing to be penetrable by air. I will not stay to describe the various ingenious appliances which M.M. Pechiney et Cie. have successively devised for the performance of the operation of treating by heat and air mixtures of manganese chloride and manganese oxide; but will only add that I believe that they have now completely overcome all the many difficulties which for a long time



seemed to render the industrial realization of that operation almost hopeless, and that it now seems certain that they will soon be manufacturing chlorine from the hydrochloric acid of the Leblanc process at the rate of the quantity of chlorine corresponding to a ton of bleaching-powder per little more than four-hundredweight of salt.

Given this result: a result diminishing by two-thirds the quantity of Leblanc soda necessary to be manufactured in order to the manufacture of a given quantity of chlorine from Leblanc hydrochloric acid: will it enable the Leblanc process, thus shorn of its proportions, to continue to live, in face of the decomposition by magnesia of the ammonium chloride of the ammonia process, and the direct obtainment from the resulting magnesium chloride of one-half of its chlorine in the free state? I trust that this may be so; but, as yet, the data for a conclusion on the point are insufficient, and of those of them which as yet exist, some tell one way, and some the other. The manufacture of chlorine from magnesium chloride, obtained by decomposing by magnesia the ammonium chloride of the ammonia process, involves much more evaporation than is required for the manufacture of the same quantity of chlorine from hydrochloric acid by the process to which I have just referred; and the whole of the chlorine obtained by the former process is diluted by other gases, whereas one-third of the chlorine obtained by the latter process is undiluted. On the other hand, the degree of dilution of the chlorine obtained from magnesium chloride is somewhat less than that of the chlorine obtained from manganese chloride; and to decompose magnesium chloride, or rather magnesium oxychloride, by heat and air, is somewhat easier than to decompose manganese chloride by the same means. All that is yet clear in the matter therefore is that chlorine will be manufactured in connection with the ammonia process, though perhaps not to any appreciable extent for a few years yet. But the question which, in face of that certainty, it is impossible not to ask: the question of whether it is or is not likely that the greatest chemical invention of the eighteenth century, if not indeed of any century, shall have ceased, by the end of the nineteenth, to be anything more than a tradition and a name: as yet it is equally impossible to answer.

If, however, unfortunately, that question shall be eventually answered in a sense adverse to those whose capital is invested in the Leblanc process: must the manufacture of soda disappear from Tyne-side? It was on Tyne-side that "artificial soda," as it used to be called, was first manufactured in England: must, in such case, this district know that industry no more? I am not of that opinion. At present, the manufacturer of ammonia soda from brine employs, per ton of soda manufactured, from a ton and a quarter to a ton and a half of coal, and the quantity of brine containing about forty-six hundredweight of salt. His coal consumption is thus light, but the quantity of salt which he consumes is twice the quantity equivalent to the soda he manufactures. Any method, however, of obtaining, either as free chlorine or as hydrochloric acid, the chlorine of the salt decomposed by the ammonia process, must necessarily involve the evaporation of all the water employed in that process,—during which evaporation that portion of the salt employed in it which at present escapes utilization will be recovered,—and must moreover involve a subsequent operation, also requiring an expenditure of fuel. Any such method must thus reduce to absolutely its theoretical amount the quantity of salt consumed in the ammonia process, per unit of soda manufactured thereby, reducing it to one-half the quantity at present required by the manufacturer of

ammonia soda from brine, but must at the same time increase his consumption of coal, per unit of soda, by at least between one hundred and two hundred per cent. Under such changed conditions: at any rate when Cleveland salt shall have become obtainable here at a cost not much greater than that of Cheshire salt in Lancashire: I think it ought to be possible to manufacture ammonia soda in this district very nearly if not quite as cheaply as in any other locality whatever.

It is not willingly that I have announced a new—and much more serious than any previous—menace to an industry already sorely tried. How much I should have preferred to have been able to prophesy smooth things with respect to the Leblanc process, no words can say. Facts, however, must be looked in the face. Nothing can be less wholesome than the atmosphere of a fools' paradise. And the watchdog at the gate, who gives timely notice of the approach of unwelcome visitors, at least renders such service as is in his power.

But if such changes as that which I have indicated as now certain to be accomplished at least partially are unwelcome, it is only in so far as they may affect individuals. By such changes, individuals may, and alas! do suffer; but the world gains by them. No industrial process is ever superseded, however partially, excepting by a better process. And in so far as the Leblanc process may be doomed to still further restriction, it can only be because some other process can give mankind cheaper soda and cheaper chlorine than it can give.

And the various proposed new processes of which I have spoken all comply with at least one of the ideals towards which all progress in such matters must henceforth tend. They all possess in common one feature which will certainly be held essential to all industrial chemical processes in a not distant future. They are all processes yielding no waste product. They are all processes completely utilizing all the constituents of all their raw materials. Excepting coal and water, nothing would be manipulated in any of them which was not finally yielded either as commercial commodity, or as regenerated reagent for use again. That way certainly lies the future.

Amongst the many transcendent merits of the beneficent invention of Nicolas Leblanc, of immortal memory, that of yielding no waste product is not included. There are spots on the sun; and the Leblanc process yields alkali-waste. Countless have been the efforts to utilize at least one of the constituents of that residue; but none of them have fully succeeded. Now, however, ninety-seven years after the invention of the process to which we chiefly owe that soap, and glass, and paper, and countless other commodities only less useful than these, have been placed within the reach of all mankind; seventy-four years after its inventor, "the creator," as Hofmann puts it, "of incalculable wealth for his species," put an end to his existence because he wanted bread, which makes it so pleasant to know that two generations afterwards he at least received a stone—statue, towards the erection of which, however, I have yet to learn that any English soda maker contributed: now, when one cannot but fear that the long day of the Leblanc process may be tending towards its close, a means of utilizing both the principal constituents of that residue seems almost in sight. To speak now of the recovery of sulphur from Leblanc soda waste may seem a kind of anti-climax; but the gallant attempt to realize the Schaffner and Helbig process to that end, upon which Mr. Alexander Chance recently expended so much enthusiastic effort, and the firm of which he is a member the greater portion of £10,000, has not only resulted in a reduction in the price of pyrites, but

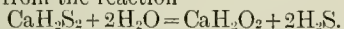
also in the suggestion of various other processes for the accomplishment of the same object : to a conceivable combination of certain parts of some of which I would ask permission to devote a very few concluding sentences.

The combined process in question would commence by bringing the calcium sulphide of alkali waste into solution as calcium sulphhydrate, by treating a mixture of waste and water by sulphuretted hydrogen. The second operation would consist in evaporating the resulting solution of calcium sulphhydrate, and so driving off its sulphur as pure  $\text{H}_2\text{S}$ ; and the third and final operation in burning off the hydrogen only of that sulphuretted hydrogen, setting its sulphur free.

The third of these operations has for some time past been under experiment in the works of Messrs. Chance Brothers, at Oldbury. The apparatus used in it is one invented by Mr. C. F. Claus, of London, and consists of a kiln in which there is a deep bed of fragments of porous material, through which, when the apparatus is at work, there is passed a mixture of sulphuretted hydrogen with a carefully regulated quantity of air. The bed of porous material is maintained at a high temperature by the heat of the reaction which takes place as the mixture of gases passes through it, and from the mixture of gases and vapours passing off from it sulphur condenses as that mixture cools. The operation thus costs nothing for fuel, and its cost for labour is scarcely more than the cost of from time to time removing sulphur from the receptacles in which it has condensed and collected.

The first of the three operations is in industrial operation at Rassuen, in the south of France, in connection with a most ingenious process, invented by M. Lombard, of Marseilles, in which calcium sulphhydrate is used to precipitate dicalcic phosphate from the solution obtained by dissolving native calcium phosphate in hydrochloric acid. It is also in operation, on a semi-industrial scale, in the alkali work of Hruschau, in Moravia. Its cost is simply that of charging waste and water into horizontal cylinders furnished with mechanical agitators, keeping these agitators in motion during the operation, and at the end of the operation discharging the contents of the cylinders.

The second of the three operations is under experiment at Hruschau, by Dr. Heinrich von Miller and Herr Opl. This second operation is obviously more costly than the first or third; but it yields, in addition to sulphuretted hydrogen, another product, which other product, it is hoped, may pay nearly if not quite the whole cost of the three operations. This other product is chemically pure calcium hydrate : resulting from the reaction—



Unlike the recovered calcium carbonate of the Schaffner and Helbig process, this calcium hydrate can be readily obtained absolutely free from impurities, since no impurities precipitate with it, and its physical condition is such that water or a solution runs from a heap of it almost as readily as from so much sand. It is incapable of causticising solutions of sodium carbonate, more than partially; it is too dense for use in Weldon oxidizers : but it seems not unlikely to prove suitable for use in bleaching powder chambers.

If it should prove to be the case, on the one hand, that this dense crystalline calcium hydrate shall have the value of an equivalent quantity of ordinary lime, and, on the other hand, that the hydrogen of sulphuretted hydrogen can be oxidized, practically completely, without at the same time oxidizing any appreciable proportion of its sulphur: then I think that an almost ideally perfect solution of the problem

of the utilization of the residual product of the Leblanc process will at last have been reached. If this should be so, one can only hope that that solution will not have arrived too late.

Mr. ISAAC LOWTHIAN BELL said he had been requested by their first president, Professor Roscoe, whose distinguished services, they were all glad, had been recognised recently by her Majesty, to thank, on their behalf, the President for the address he had just delivered; and, as that was the last meeting at which their President, Mr. Weldon, would preside, he would take that opportunity of combining with those thanks the thanks which were his due for the very distinguished manner in which he had discharged the duties of his high office. These duties had been of such a character that it would require all the merits of his successor to reconcile them to his loss as their President. They could only hope that in his more retired sphere he would continue to address them in some of those friendly "barks" of which he had given them so excellent an example on the present occasion. He could only regret, for his own part, that while the President warned them of the approach of the most unwelcome intruder, he himself, upon more than one occasion—perhaps upon the present occasion—was the intruder himself. His friend the President had spoken to them almost in terms of apology for the reduced production of Leblanc soda. He (Mr. Bell) was one of the transgressors in that particular line. In the iron trade, as they all knew, they had been compelled, before the soda trade followed their example, to adopt a similar line of conduct. And he had always excused himself upon this ground: He had supposed, merely, of course, for the sake of argument, that he was the only iron manufacturer in the kingdom. Surely, if, after the lapse of several years, he found that his capacity for making iron was much more than was required, no one whose opinion was worth listening to, he thought, would maintain that he was acting otherwise than most prudently in suiting the supply to the demand. He submitted that that which was proper and advisable on the part of one individual was equally applicable to the whole trade. They had had a very excellent and eloquent retrospect of the soda trade; they had had an equally eloquent forecast of what was probably to come. What he would like to know was, when were they to rest and be thankful? when to cease being overwhelmed with new processes with which Mr. Weldon might choose to associate his name, as he had done in the past? Mr. Weldon consoled them with the statement that while they suffered themselves it was for the gain of the world. He had no doubt his friends in the soda trade were philosophers enough to allow that consolation to have its proper weight. He must say that if they could derive any satisfaction, it must be that which they had received that morning in listening to the language of Mr. Weldon, of being assured, as they had just been, that they had done much for the prosperity, not only of this district, but of the world at large. He moved a vote of thanks to the President for his address.

Professor ROSCOE seconded the vote of thanks. Speaking in the name of those who came from a distance, he said that their journey to Newcastle had already been repaid by their having heard what they had in that excellent address. For his own part, he happened to know some of the duties and some of the work devolving upon the President of that Society, and he could only say that they owed Mr. Weldon a deep debt of gratitude. The success which had attended that Society was in a great measure due to the earnest efforts which he, during the last twelve months, had made to put the Society on a proper footing.



The PRESIDENT, in replying, said he felt very proud to have held the honourable position of President of that Society, and he regretted that circumstances had prevented him from taking advantage as he had so wished to do of all the opportunities of usefulness which it presented. Still, he might claim to have emulated the example set him by all his colleagues, and by absolutely every officer of the Society, without exception, in such devotion to the interests of the Society as was possible to him.

#### ALTERATION OF BYE-LAWS.

Continuing, the President said there were certain alterations proposed in the bye-laws of the Society. As the bye-laws stood at present, the only subject they had power to discuss was that of improvements in the various branches of chemical manufacture; and he thought no straining of these words could make them include, for example, the discussion of modes of sampling and testing, in which so many of their members had taken so much interest. It was perfectly clear that by their bye-laws, as they at present stood, they were debarred from discussing many subjects which it would be useful that they should be able to discuss. The Council therefore recommended that that meeting should confer upon the Society power to discuss all matters whatever of interest in connection with the practice of applied chemistry. There were other changes proposed by the Council, one making the retiring president eligible for re-election, and another removing all restriction with respect to the local sections.—These amendments, with others of a minor character, were approved of and confirmed.

#### ELECTION OF PRESIDENT AND COUNCIL.

On the motion of Mr. A. M. CHANCE, seconded by Mr. TYRER, Mr. J. F. STARK and Prof. HUNTINGTON were appointed scrutators of the ballot papers sent in, and the result was the election of President and the Members of Council as set forth in the list, printed on the first page of the present issue of this Journal.

#### NEXT PLACE OF MEETING.

Mr. LUDWIG MOND then moved that the Annual Meeting of the Society be held next year at London, where it was their custom to hold every second meeting.—Mr. NEWALL, of Gateshead, seconded the motion, which was agreed to.

#### UNIFORM METHODS OF ANALYSIS AND TESTING.

Mr. TYRER moved—

That it is desirable to appoint a standing committee to consider how to carry into practical effect the resolutions of the various local Sections upon uniform methods of analysis and testing. The Committee to consist of the Executive Officers of the various local sections—two representatives from each Section—and such other experts as the Committee may, from time to time, nominate. This Committee to report to the Council.

Mr. WILLIAMS seconded the resolution, which was carried unanimously.

#### MODIFICATION OF BYE-LAWS.

It was resolved, on the motion of Mr. TYRER, seconded by Mr. A. M. CHANCE—

That the modifications of the Bye-laws which are set forth in the May Number of the Journal be, and hereby are accepted and adopted by this meeting, with the following exceptions and amendments, viz:

That Rule 18 be amended by adding thereto the words "No Member shall sign more than one Nomination Form."

That Rule 19 be amended by deleting the whole of second clause thereof.

That Rule 20 be amended by inserting after the word "and" in the second clause thereof the words "the names of all members," and the deletion of the words "the persons."

That as a consequence Rule 22 to read—

Any member shall be at liberty to strike out any name or names of ordinary members of Council printed in his voting paper, provided that the names so struck out are those in excess of the number of vacancies to be filled up.

That Rules 48 and 50 be amended by the insertion of the word "advertising" after the word "stationery" in the first line thereof.

#### THE ANNUAL DINNER.

The Annual Dinner of the members of the Society of Chemical Industry, was held in the Banqueting Hall, Jesmond. Nearly 180 members were present, besides some visitors. The president (Mr. Weldon, F.R.S.), occupied the chair, and he was supported by the Mayor of Newcastle (Dr. Newton) and Sheriff (Mr. Nelson), Professor Roscoe, Dr. Perkin Mr. Ludwig Mond, Mr. J. T. Dunn, Alderman Cail, Mr. T. Tyrer, Mr. Lowthian Bell, and Mr. A. M. Chance.

The CHAIRMAN proposed the toast of "The Queen, the Prince and Princess of Wales and the rest of the Royal Family," which was drunk with the usual honours.

The CHAIRMAN, in calling upon Professor Roscoe to propose the next toast, said he could not do so without expressing his own great gratification—a gratification which they would all heartily share—at the Royal recognition of Professor Roscoe's great services, not only to science, but also to the cause of education, which her Majesty had been graciously pleased to signify her intention of bestowing upon him. For his own part, he was afraid that "Sir Henry Roscoe" would never sound quite so gratefully in his ears as the older title under which their first president had won his great place in science, and in the affectionate regard of all who had the privilege of knowing him. Still they could not but rejoice at the honour which he was about to receive from the highest fountain of honour; and although he would soon cease to be plain Professor Roscoe, yet in gaining his new title he would not lose his old one, but the two would combine together to form that compound of which Mr. Isaac Lowthian Bell had spoken in the morning, "Professor Sir Henry Roscoe."

Professor ROSCOE then proposed "Success to the Industries of Newcastle." He was sure, he said, they would excuse him if he only very briefly alluded to the circumstances to which their President had drawn their attention. He felt most deeply the too flattering terms in which their President had spoken of his merits, and the kind way in which his friends, the members of the Society of Chemical Industry, had received the notification. He wished to say also that he felt it was a recognition less of his own merits than of the profession of science, and in that respect he received it gratefully and thankfully. He had the honour to propose to them a toast which he was sure would receive not only their attention but would be drunk with high honours. It was, "The Industries of Newcastle." He had to call upon them to wish well to those great industries which were founded by Losh and by Pattinson, and were yet of world wide renown. He supposed that here, as everywhere else, there was room for improvement still, and he wished them well. He wished to thank most heartily those gentlemen who had kindly placed their works at the disposal of the society on their present visit, and, first of all, he had to thank Sir William Armstrong for having, in the most kind way, placed that noble hall at their disposal. They had also to thank Sir William Armstrong for allowing them to see through that magnificent series of works that stretched along the Tyne, and where those great engines of war were manufactured. It would be too long a list were he to mention the names of all the gentlemen who had opened their works to them on that occasion, and he could only thank them one and all. He coupled the toast with the names of two gentlemen well known not only there, and not only in the Society of Chemical Industry, but everywhere where applied chemistry was known—Mr. Hugh Lee Pattinson and Mr. Alfred Allhusen.

Mr. PATTINSON, in responding, said he begged to thank them on the part of the "distressed" industries of the Tyne for the very flattering and kind manner in which they had proposed their prosperity. It needed all their good wishes to revive the industries of the Tyne from the state of desolation and deep despair to which they had descended. They had been called upon in the days of desolation for the songs of Zion, and they responded to the call. The Northern chemical manufacturers would never give way. They had seen bad days and good days, and, like men of mettle, they bore the days of prosperity with calmness and moderation, and did not sink under the days of adversity. It was certainly an unkind cut to call upon one of the most depressed of the Tyne industries to respond, after the terrible flagellation that the President had inflicted upon them that morning. But that flagellation was inflicted by a friendly hand, and they knew there was no venom in the prognostication. Threatened men live long. They had heard of their extinction a good many years ago, yet they survived. They therefore did not despair, but depended upon that celebrated expression of a very great statesman, "The resources of civilization are not exhausted." They might be threatened by some terrific invention by Mr. Mond, M. Pechiney, and other great *savants*, but in their own works might arise some great inventor, so that with the poison might be administered the antidote. The industries of the Tyne were really in a "badish" way: lead, coal, timber—anything they might choose to name—were all in the ditch together, and that was one little consolation to him personally. However, as long as chlorine was required in the industries of the world, there would be room for their existence; as long as it held its position as one of the elemental bodies of chemistry, they had a good prospect before them. It was valuable and always would be valuable, just in the same way as gold and diamonds were always valuable. He thanked then on behalf of the depressed and distressed industries of the Tyne for the toast, and hoped they would emerge from their present distress to prosper as they had done before.

Mr. ALFRED ALLHUSEN, who also responded, said he endorsed every word that Mr. Pattinson had said. He had, he believed, conveyed the feelings of the majority of the Leblanc soda makers on the Tyne. He hoped that the members of the Society would find some pleasure in visiting the various works on the Tyne. He thanked them for so heartily drinking the toast of "The Industries of the Tyne."

The CHAIRMAN then rose to propose "The Society of Chemical Industry," and was received with loud applause. He said his duty would be an extremely light one, for, in proposing that toast to that assembly, to make a speech in recommendation of it was as unnecessary, and would be almost as impertinent, as to offer argument in support of what were called the usual loyal toasts. With very few exceptions, all present were members of the Society of Chemical Industry, and they would not have been such if they had not believed that the objects for which that Society was established were useful and important objects; and they, therefore, needed no persuasion to drink to its increased prosperity and continued success. He had infinite satisfaction in associating with that toast the name of the famous chemist and illustrious inventor who had been that morning elected President of their Society—Dr. Perkin.

Dr. PERKIN, in rising to respond, was received with acclamation. He said he had to thank the president for having coupled his name with the toast, and to thank also his fellow members for the hearty manner in which they had received the toast. He also had to thank them for the great honour they had done him

in electing him President for the next year. He felt it was a position which he would not be very well able to fill, after the eminent men who had already occupied it. He thanked the chairman very much for the very kind and flattering remarks he had made in reference to his (the speaker's) past career. It was very satisfactory to find that the Society, although so young, had become so large. He very well remembered how, four years ago, when Professor Roscoe asked him to second the proposition that that Society should be formed, that he did so with some misgivings. He assumed now that the experiment had been a greater success even than Professor Roscoe anticipated. The success of that Society showed that it was needed; and there could be no doubt that the meeting together of men to work out chemistry as applied to manufactures, and the interchange of thought which must as a consequence accrue, must have a stimulating influence and do good to the advancement of the industries of this country. They had heard from Mr. Weldon that day all about the chemical industries with reference to the soda process and the great changes that had taken place. The manufacture nowadays was a very different thing from what it was five-and-twenty or thirty years ago. Changes then were comparatively slow, and now they were rapid—he might almost say incessant. And for a man to be a successful manufacturer now, he required to be ever on the march, and constantly pressing forward. For their Society he thought there was a great hope for the future. In the meantime, they must be very thorough, and they must not be standing still, but pressing forward. Their cry must be always "Excelsior! Excelsior!"

The CHAIRMAN proposed "The Officers of the Society," and coupled with the toast the names of their Honorary Foreign Secretary, Mr. Ludwig Mond; the Editor of their Journal, Mr. Watson Smith; and that of their General Secretary, Mr. Cresswell.

Mr. LUDWIG MOND responded.

Mr. WATSON SMITH, said, as Editor of their Journal and as a teacher of applied chemistry, that from the time when he first sketched out a plan of the Journal for the Council of the infant Society in 1881, it had been his ambition and desire that their Journal should form a kind of text-book in applied chemistry for this country, and that, annually bound, its volumes should serve as a work of reference analogous to the great German *Jahresbericht* of Dr. Fischer.

Their competitive friends across the channel seemed to see some tangible advantage in joining their ranks and taking the Journal.

The Editor of their Journal owed more, both personally and editorially, to the retiring President, Mr. Weldon, than he could adequately express in words. Mr. Weldon was a veteran journalist and editor, and what was more, a practical logician as well as a practical chemist, and the continued contact with so experienced and willing a friend and guide during the last twelve months could not but be of the highest benefit to a younger and less experienced man.

After the address of this morning, someone expressed despair at the almost illimitable prospect of change and revolution in certain of their industries. How was this danger to be provided for? What could be done to cope with it? The key to the riddle was not far to seek, and if those who wanted it would now turn to the recent closing address of the session, delivered to the Chemical Society by their new President, they would find it there. Thus, their retiring President suggested a riddle, anticipated and solved by their new President, Dr. Perkin.

Mr. CRESSWELL said he had only helped in a



humble way towards the elevation of a super-structure, the foundation of which had been laid by others. The men who had gone before had not such an encouraging task as he had, and he was sure in remembering him they had remembered their past Honorary Secretary, Mr. Davis.

The CHAIRMAN said they had with them that evening two eminent Frenchmen—Monsieur Kolb, whose researches made some thirty years ago upon the chemistry of the Leblanc process they were all acquainted with, and Monsieur Lamy, a son of the eminent French discoverer of *thallium*. Their editor had referred to their competitors across the channel. M. Kolb was one of these, but he was a friendly competitor. He begged to propose his health and that of M. Lamy.

M. KOLB responded in French, and concluded by proposing the toast of "The Ladies," which was drunk with musical honours.

Mr. T. TYRER (Secretary of the London Section) proposed "The Newcastle Section." He was sure that at their present meeting, which had been so pleasant and so comfortable, the arrangements had been very efficient, and there was not likely to follow any of that disappointment apt to be created on such occasions. In this case the onus of the arrangements had fallen chiefly upon the shoulders of Mr. J. T. Dunn, whose name he had great pleasure in coupling with the toast he had proposed.

Mr. J. T. DUNN, who was received with applause, said he had only to thank them in the name of the committee for the manner in which they had responded to the toast, and to say that the committee would feel themselves amply rewarded for any trouble which they had taken to make the arrangements for the meeting, if these arrangements passed off satisfactorily. The large number of members who had assembled there at the meeting almost exceeded their expectations, and would leave pleasant impressions behind them. Their chairman, Mr. J. C. Stevenson, was, unfortunately, engaged on a Parliamentary committee, but he hoped to be able to leave his duties there in time to reach home that evening, and he hoped to join them on the day after. Although the Society of Chemical Industry would always associate foundation with Manchester, and more particularly with the name of their first president (Professor Roscoe), he thought that in this (the Newcastle Section) they would always prefer rather to claim as their founders two names which he would recall to them, Mr. Robert C. Clapham and Professor Algernon Freire-Marreco. These two men were the instruments of the foundation of the old society, and, singularly enough they died within a month or two of each other, only three or four months before the old society, in a newer birth, merged its own existence in the larger existence of the Society of Chemical Industry.

Mr. A. M. CHANCE proposed "The Visitors," to which toast the MAYOR and SHERIFF responded.

During the evening an excellent selection of vocal music was sung by a number of gentlemen, Members of the Society. Mr. Tyrer greatly entertained the company by singing a second new version of "Ye Leather Bottel."

#### VISITS TO WORKS AND FACTORIES.

The first visit was paid early in the afternoon of Wednesday, the 9th inst., to the office of the

#### "NEWCASTLE DAILY AND WEEKLY CHRONICLE."

The party was received by Mr. R. B. Reed, by whom the members were conducted to the composing depart-

ment. One of the page-formes of the mid-day edition of the *Daily Chronicle* was opened, and into it was inserted a brief account of the visit of the party. The process of stereotyping the matter was then gone through, an impression being taken on a prepared matrix, from which several castings of metal were taken and made ready for the printing machines. In the machine department, the printing process was next witnessed, and each visitor was supplied with a copy of the *Weekly Chronicle* of the previous Saturday, containing a short account of the retiring President, Mr. Weldon, F.R.S., with an excellent portrait, and one of the mid-day edition of the *Daily Chronicle*, with an account of the visit.

#### THE ELSWICK ENGINEERING WORKS

(Sir Wm. G. Armstrong, Mitchell, & Co., Limited) were next visited, about two hundred of the visitors journeying up the river Tyne for the purpose in one of the Tyne General Ferry Company's Steamers at 2:45 p.m. to High Elswick.

#### THE ST. ANTHONY'S LEAD WORKS

(Messrs. Locke, Blakett, and Co.) On Thursday, the 10th, a party of about two hundred members left the Quay Side by the steamer for the above works. At St. Anthony's, Mr. J. Warwick met the party, and led the way to the factory, where the manufacture of white lead by Milner's process, of red lead, of ground and flake litharge, and of lead in the form of sheet, pipes, &c., was witnessed. The visitors were also shown the rolling of sheet lead, the making of pipe lead, the desilvering of pig-lead by the zinc process, which excited much interest, and the production of red lead and of white lead by a new process. The party then re-embarked, and proceeded up the Tyne to the

#### NEWCASTLE CHEMICAL WORKS,

where they were met by Mr. Alfred Allhusen, who conducted them through the cooperage, the sulphuric acid department, the salt-cake department, the refining, bleaching powder, and other departments. The works, commenced in 1834, and transferred to the limited company in 1872, are now the most extensive on the Tyne. They cover an area of about 62 acres, burn annually about 30,000 tons of pyrites, decompose some 50,000 tons of salt, and produce about 72,000 tons of finished products—alkali, soda crystals, and bleaching powder. The number of men employed is about 1,000. Pyrites are burnt in Schaffner and Helbig's shelf kilns, there being 129 of these in the department. The hand furnaces in the works still linger, but one of Jones's mechanical furnaces, heated by gas producers (which also heat part of the boilers), is efficiently at work, decomposing 300 tons of salt per week, and was shown in operation, whilst two others of modified form, and of very strong build, are in course of construction. At the invitation of Messrs. Allhusen, the entire company partook of refreshments in a large sulphuric acid chamber which was in course of being rebuilt, and in another chamber they were photographed.

#### PALMER'S SHIPBUILDING WORKS AND IRON CO.

at Jarrow formed the object of the last visit of the day. Here, under the guidance of Mr. P. A. Berkeley the visitors were shown first the blast furnaces, and one of the three operative furnaces was tapped; then rolling mills, where sheets and plates were being rolled and cut; the boiler shop, the engine works, and finally the shipyard. The party returned to Newcastle by boat.

On Friday the members first visited

### THE SWING BRIDGE

over the Tyne, and afterwards obtained a fine view of it in operation from the steamer as they proceeded to the works of the

### THARSIS SULPHUR AND COPPER COMPANY.

At these works, which have been in operation about twenty years, they saw the process of the extraction of copper from burnt pyrites. This residue, treated with salt and calcined, and afterwards lixiviated in tanks, is treated for gold and silver, and then passes into vessels filled with scrap iron. The copper precipitate obtained is then taken to the smelting furnaces. The copper is chiefly sold in ingots, cakes, and wire bars. Messrs. C. Tennant and Co.'s Alkali Works were next visited. These were commenced in 1865, on a much smaller scale than now, and were worked as a branch of the older establishment at St. Rollox, Glasgow, where the manufacture of bleaching powder was first introduced into this country. The products are bleaching powder and soda, the total made at Hebburn being about 800 tons weekly. One of the special features of the works is that four of Mactear's salt-cake furnaces decompose the whole of the salt, hand labour having been abolished. About 700 men are employed in the various departments.

Lunch was served in the Drill Hall, the arrangements being under the charge of Mr. J. McCulloch.

There were no formal toasts, but Professor Roscoe proposed in a few sentences the thanks of the Society to the gentlemen who had so kindly thrown open their works to the visitors. This was seconded by Mr. J. C. Stephenson, M.P.

### THE LEAD WORKS OF MESSRS. COOKSON AND CO.

were next visited. An exhibit of the raw materials and finished products of the works was shown, prominent being a block of silver computed to be worth from £2,200 to £2,300, and weighing over 6cwt. The works are for the smelting of lead and its desilverising; and it was stated that the furnaces when fully charged would hold 1,200 tons of lead.

After this visit, the party proceeded by the special steamer placed at the disposal of the Committee by the Tyne Commission, to the North Pier, Tynemouth, where its members separated.

Another excursion by members of the Society was made to the coke ovens at South Medomsley, where the

### JAMESON COKING PROCESS

was seen in operation; and also to Crook to see the extensive set of coke ovens on the Simon-Carvès principle at

### MESSRS. PEASE AND PARTNER'S WEST COLLIERIES.

The production of good metallurgical coke, with coal tar, and ammoniacal liquor, equal and similar to that furnished in any gas-works, was here witnessed.

### CONVERSAZIONE AND RECEPTION.

On Thursday evening a reception was held in the Assembly Rooms, Westgate Road, by Mr. Walter Weldon, F.R.S. (retiring President of the Society of Chemical Industry), at eight o'clock. A most enjoyable and admirably conducted musical entertainment was given in the large room by a string orchestra and a chorus of men's voices (The "Antigone" Amateur Male-voice Choir), under the leadership of Mr. J. H. Amer. A selection from Mendelssohn's *Antigone* amongst other things, was given most effectively by the above-named excellent *Männerchor*, a kind of performance too seldom heard in this country. Refreshments were served on a large and liberal scale in the hall. The entertainment was a great success. The attendance of ladies and gentlemen

was very large, and the enjoyment of the evening rendered brilliant testimony to the indefatigable exertions of the Local Secretary (Mr. J. T. Dunn), and the Local Committee.

The Newcastle meetings as a whole have been pronounced among the most successful, not only as regards their general interest, but as regards the large attendance, the general goodfellowship created, and numerous new acquaintances formed amongst the members of the Society—surely one of the chief objects of meetings of this kind.

The Annual Meeting of the Society of Chemical Industry in July, 1884, will live long in the recollection of most members present, as a brief bright reminiscence in which many known to each other only by reputation, now came face to face, and engaged in friendly converse.

## London Section.

Chairman: David Howard.

### Committee:

Sir F. A. Abel.	B. E. R. Newlands
H. E. Armstrong.	B. Redwood.
W. Crowder.	T. Royle.
C. Graham.	John Spiller.
S. Hall.	G. C. Trewby.
A. K. Huntington.	W. Weldon.
R. Messel.	J. Williams.

Local Sec.: Thos. Tyrer, Garden Wharf, Church Road, Battersea, W.

CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE  
Monday, May 5th, 1884.

### MR. DAVID HOWARD IN THE CHAIR.

## ON THE PROCESSES CONCERNED IN THE CONVERSION OF STARCH INTO ALCOHOL, AND THEIR RELATION TO BREWING AND DISTILLING.

BY W. S. SQUIRE, PH.D.

It is only in the last few years that the processes concerned in the conversion of starch into alcohol have been scientifically investigated. Owing to the complicated nature of the influences at work, experimenters are far from unanimous in the conclusions at which they have arrived. Still, many important facts have been elicited, and I thought that a short sketch of our present knowledge of this subject might interest the members of this society. Where there is a conflict of evidence, I have ventured to select that which is in accordance with my own experiments and practice, without at all wishing to imply that my own experiments are more trustworthy than those of others, or that they by any means set disputed questions at rest. Those who are bold enough to address the members of a society like this, must be prepared for a pretty severe cross-examination at the end of their discourse, and for that reason, if for no other, I must be prepared to vouch personally for the facts I adduce in support of the conclusions I venture to draw.

As is the case with all ancient industries, considerable perfection in the processes employed in the conversion of starch into alcohol has been arrived at in the course of ages by pure empiricism. The practical man, warned by failures in one direction or another, learnt by sheer practice that method of working which yielded him the best results, without troubling himself about the why and the wherefore. He knew what temperature was, at certain stages, of importance, but



he judged of this at first, either by the amount of steam coming off from his vessel, or by dipping his finger into the hot liquor. For a long time he sneered at the thermometer as a new-fangled concern unworthy of a practical man who knew his business, and was guided in his operations by all sorts of rough-and-ready rules and appearances, for which he had the most fantastical explanations, but more generally no explanation at all. In fact, the brewers and distillers may be said to have formed a close corporation of rule-of-thumbists of the first order.

As ignorance and credulity go ever hand in hand, so it was, and to a certain extent still is, in this great industry. Secrets which are no secrets at all are jealously guarded. Recipes for setting bad beer right, and for fortifying and tinkering up yeast, all perfectly worthless, are bought and sold, and there is scarcely any industry in which mystery-mongering and quackery do so good a trade.

It is curious how professors of the art of brewing hang on to these electrical notions. A book published in the last few days by a well-known author\*, contains this passage, at page 63. Speaking of fermenting vessels the author says: "We are apt to have far too many metal fittings, and the circular coils of attemperating piping, with brass parachute pipe passing down the centre line forcibly reminds one of a Leyden jar arrangement, and in many instances seems to be one." Then, in a foot note, he describes a Leyden jar, and says, "in the arrangement spoken of, the parachute pipe would be the inner coat, the attemperating coils the outer, and the layer of beer between them, the intervening glass!"

I have here another book†. The author is a practical brewer, hailing from Burton-on-Trent. He says page 30, "that acetous fermentation will occur in the mash-tun from the application of mashing heats taken at too low degrees of heat, whereby the pores of the liquor are so much opened that they readily imbibe too large a portion of atmospheric air. Acetous fermentation also ensues when the mashing heats are taken too high, thereby in a great measure closing the pores of the worts to the fermentation matter or yeast."

I have here a book of much greater value‡—at least it is published at 10 guineas. He says at page 69: "Pasteur's discoveries, proving that air-borne germs are the cause of what was supposed to be spontaneous germination, although of the highest importance in surgery and general medical practice, are of little use to the brewer. If the malt has been properly cured and brewed, the conditions of life for these air-borne creatures are not in the wort, and the wort is safe." "Pasteur's work shews yeast cells in various conditions, and were he or others turning the microscope to the cleavages or granules of fractured malt, they might be able to detect and check in time the causes of these abnormal conditions in yeast." It seems from this that Pasteur has been using his microscope to small purpose.

As might be supposed, the author speaks with profound disdain of the Bavarian system of brewing. He says, "The Bavarians, as well as the Germans generally, are, to the mind of the author, quite astray, both with their malt-drying, and their mashing processes." "The author has conversed with German brewers, and seen the process on a large scale, and he believes these people would be glad to exchange their system

for ours." The German system is, in his opinion, "the result of notions of cleanliness following on loss of tradition, or perhaps of mere loose drifting about because of never having had proper charts for guidance." That cold has anything to do with bottom fermentation he positively denies. I could multiply instances, but enough has been said to shew the sort of literature offered, and, what is more, eagerly bought at fancy prices, by the brewing trade.

Distillers and brewers are, in the widest sense of the term, chemical manufacturers, but the number of those who have any notion of the chemical changes going on in their mash tuns and fermenting backs is extremely limited. I am not speaking of those great brewers who employ the highest scientific skill, but of brewers generally.

It must not, however, be supposed that empiricism had gone very far wrong. When science stepped in to investigate she could only confirm much of the experience of ages, and admit that practice was in many respects right. It generally happens so. Agriculturists had found out the value of farm-yard manure, before Liebig and Gilbert pointed out the constituents to the presence of which in the manure its value is due; and washerwomen spat on their flat-irons to ascertain the heat by the flying off of the saliva, long before the days of Leidenfrost.

As the subject is a very wide one, and the time at my disposal is limited, I propose to confine myself, on the present occasion, to the action of organic and organised ferments, reserving for a future communication those conversions which are effected by other means. For the same reason I cannot deal with the conversion of barley and other seeds into malt, but will assume the malting process to be completed.

Our starting point, starch, is a body of very complicated structure, and probably of very complicated composition. It occurs widely distributed through the vegetable kingdom, and consists of minute grains which differ greatly in size and form, according to the vegetable source from which they have been derived. The starch granules are so characteristic of the plants which furnish them, that when vegetable powders are examined under the microscope, the form and size of the starch granules (if any be present) are often the best indication of the nature of the plant from which the powder is derived. The diameter of the grains varies from 2 to 60 micromillimeters\*. The starch granules are either simple or compound. The simple grains, when examined under the microscope, present the appearance of round, or more or less egg-shaped bodies, offering distinct evidences of concentric stratification grouped round a point which is sometimes central, and sometimes eccentric, in which latter case it is near to the small end of the granules, as for instance in potato starch. The layers are not formed by the deposition of one on the other proceeding from within outward like the rings of an exogenous tree, but the fresh deposits are formed at the centre of the grains, pushing out and condensing the previous layers, much in the same way as in the endogenous stemst. The compound grains present the appearance of rounded masses with a reticulated structure, as if minute hexagons or rather dodecahedrons of somewhat irregular shape had been agglomerated together. Portions of these compound grains are sometimes found detached, in which case they occur as angular bodies of very small size. Rice starch for example. Sometimes, as in the case of the cereal grasses, there are two kinds of starch granules, large and small. The diameters being generally as 1 to 4 or 5. The two kinds are quite

\* The Theory and Practice of Modern Brewing, by Frank Faulkner, London, F. W. Lyon, 1881.

† The Art of Brewing India Pale Ale, by James Herbert, practical brewer, Burton-upon-Trent, fifth edition, 1872, pp. 66, published by the author, price 10/6.

‡ Selection of the Practical Points of Malting and Brewing, by James Steel, pp. 123, London, E. and F. N. Spon, 1881.

\* Micromillimeter =  $\frac{1}{1000}$  Millimeter =  $\frac{1}{25400}$  inch.

† Nageli, Die Starkekörner.

distinct, no intermediate sizes occurring. (Drawings of the various starches were exhibited on the walls of the room. Of these drawings the photographic reductions are exhibited on the adjoining page.)

The starch granule when thoroughly dry is capable of absorbing from 10 to 18 per cent. of water, of which it may be again deprived without altering its physical constitution. In other respects it is not acted upon by cold water. Starch consists of two bodies, which have received the names of granulose (constituting by far the greater part of the whole), and amylocellulose. It is of the latter that the external envelope of the granule consists.

When starch granules are heated with water they begin to swell. Cracks appear in them, the envelope becomes torn, and the whole is converted into the familiar starch paste. The following table\* shows the temperature at which this change takes place in different starches:—

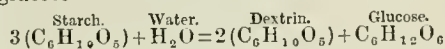
	Swelling up.	Commencement Gelatinisation.	Perfectly Gelatinised.
Rye .....	45C.	50C.	55C.
Maize .....	50	55	62·5
Barley .....	37·5	57·5	62·5
Potato .....	46	59	62·5
Rice .....	54	59	62
Wheat .....	50	65	67·5

These figures hold good for the pure starches separated from the vegetable substance to which they belong. In some cases, however, the starch *in situ* requires a much greater heat to break it up, owing to the vegetable tissue in which it is embedded protecting it from the action of water. Crushed potatoes, rice and maize require long boiling with water, even under pressure, before the starch which they contain is completely gelatinised. The starch in crushed rye, barley and wheat breaks up nearly at the temperature given. A thin starch paste may be filtered through paper, and the solution, which is perfectly clear, contains the granulose, while the amylocellulose remains on the filter in the form of an opalescent jelly. Still it is very doubtful whether the granulose in the filtrate is really in solution, or only in a fine state of suspension. When porous bodies, such as fine linen cloth, are dipped into starch paste the water only is absorbed, the amylocellulose and the bulk of the granulose being left on the surface, and this is afterwards partially converted into dextrine by the hot iron of the washerwoman. Whether this be a true state of solution or not, it is certain that when starch is heated with water under pressure for some time, it is converted into a perfectly soluble body, known as soluble starch, and this solution, on cooling, no longer gelatinises. Starch heated in glycerine also undergoes this change. The amylocellulose differs considerably from ordinary cellulose. It is not soluble in ammonia and cupric oxide, and unlike cellulose, it is attacked by diastase. The well-known reaction of iodine with starch applies to granulose in all its modifications, but amylocellulose does not give the blue colour. It is coloured reddish or yellowish.

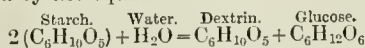
Important for us is the change which starch undergoes when acted upon by diastase. This body, which is very widely distributed in the vegetable kingdom, occurs in the largest quantity in germinating seeds, and possesses the property of converting gelatinised starch into saccharine matter. How this action is brought about, it will be convenient to study later on. For our present purpose a cold solution of malt may be regarded as a solution of diastase. This solution has no action whatever upon starch granules in the cold. The granulose of the starch is enclosed in

a membranous envelope, and as diastase is a perfect colloid it cannot get to the interior of the granule. If this envelope be broken by rubbing the starch with fine sand in a mortar, the granulose is exposed, and the diastase acts upon it even in the cold, though the granulose alone is attacked, the amylocellulose being unacted upon. Upon starch paste, diastase acts with great energy, even in the cold, though more rapidly if the temperature be raised to between 50° and 60°. In one or two minutes the paste loses its viscous character, the iodine reaction, which was at first dark blue, passes through violet to red, and in from 5 to 10 minutes disappears altogether. During this time the solution, which is quite limpid, has acquired a sweet taste.

The nature of this action has been studied by Dubrunfaut, O'Sullivan, Musculus, and Brown and Heron. Until about ten years ago it was supposed that the sugar produced from starch was grape sugar or glucose. Dubrunfaut\*, it is true, pointed out in 1847 that the sugar produced was not glucose, but a sugar "sui generis," to which he gave the name of maltose. For some reason or other, this announcement excited no attention, and the thing was forgotten. So long ago as 1812, Vogel discovered that a gummy body was produced in addition to sugar. This body was examined by Biot and Persoz†, who gave it the name of dextrin, on account of its property of rotating to the right a ray of polarised light. Up to 1860 it was supposed that this body, dextrin, was the intermediate stage in the production of glucose, the starch being first converted into dextrin, and then, by the further action of diastase, into glucose. In 1860,‡ Musculus maintained that the dextrin and glucose were formed simultaneously, the starch taking up water and splitting into dextrin and glucose—



In 1870, Schwarz§ called attention to the influence of temperature (of which, in a vague sort of way, brewers and distillers were quite aware), and shewed that at 60° (140° F.), and under, the reaction was expressed by the equation—



At temperatures above 60° he found that the action was represented by Musculus' formula.

In 1872, O'Sullivan|| rediscovered Dubrunfaut's maltose, and examined its properties. He found it to be a body totally distinct from glucose, having greater optical properties and a power of reducing from Fehling's solution only two-thirds as much cuprous oxide as is reduced by glucose. The composition of maltose was found by O'Sullivan to be  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ , and he proposed for starch  $\text{C}_{18}\text{H}_{30}\text{O}_{15}$ .

Under these circumstances the action would be represented thus:—

	Starch.	Maltose.	Dextrin.
At or below 63° (145° F.)	$\text{C}_{18}\text{H}_{30}\text{O}_{15} + \text{H}_2\text{O} = \text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{C}_6\text{H}_{10}\text{O}_5$		
Percentage.....		67·85	32·15
Temperature $\text{C}_{18}\text{H}_{30}\text{O}_{15}$			
64° to 70°	$\text{C}_{18}\text{H}_{30}\text{O}_{15} + \text{H}_2\text{O} = \text{C}_{12}\text{H}_{22}\text{O}_{11} + 4(\text{C}_6\text{H}_{10}\text{O}_5)$		
(147°-158° F.)		34·5	65·5
Percentage.....			
Temperature			
70° to 80°	$4(\text{C}_{18}\text{H}_{30}\text{O}_{15}) + \text{H}_2\text{O} = \text{C}_{12}\text{H}_{22}\text{O}_{11} + 10(\text{C}_6\text{H}_{10}\text{O}_5)$		
(158°-176° F.)		17·4	82·6
Percentage.....			
Reducing power:—	Maltose, 65; Dextro-glucose=100.		

\* Ann. Chem. Phys. [3], 21, 178.

† Ann. Chem. Phys. [2], 52, 72.

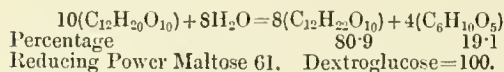
‡ Compt. rend. 54, 191.

§ Journ. f. prakt. Chemie. (1870), I., 212.

|| Journ. Chem. Soc., 1876, I., 478, and II., 25.



Brown and Heron,\* in a very elaborate investigation of this subject, find that the quantity of maltose produced is greater than that stated by O'Sullivan, and propose the following formula as expressing the normal action of diastase on gelatinised starch at 60° (140° F.) and below :—



When the reactions took place at higher temperatures the action was retarded, and the quantity of maltose produced was less. Brown and Heron's results are best shewn by the following graphic table which I copy from their paper :—

the optical power diminishes, while the cupric oxide reducing power increases until at last both correspond to the properties of pure maltose. Thus—

No. of Transformation.	Sp. Rotation.	Cu <sub>2</sub> O reduced.	Resulting Dextrin.
Soluble starch...	216.0	0	
1.....	209.0	6.4	...Erythrodextrin $\alpha$
2.....	202.2	12.7	... " $\beta$
3.....	195.4	18.9	...Achroodextrin $\alpha$
4.....	188.7	25.2	... " $\beta$
5.....	182.1	31.3	... " $\gamma$
6.....	175.6	37.3	... " $\delta$
7.....	169.0	43.3	... " $\epsilon$
8.....	162.6	49.3	... " $\zeta$
9.....	156.3	55.1	... " $\eta$
Maltose .....	150.0	61.0	—

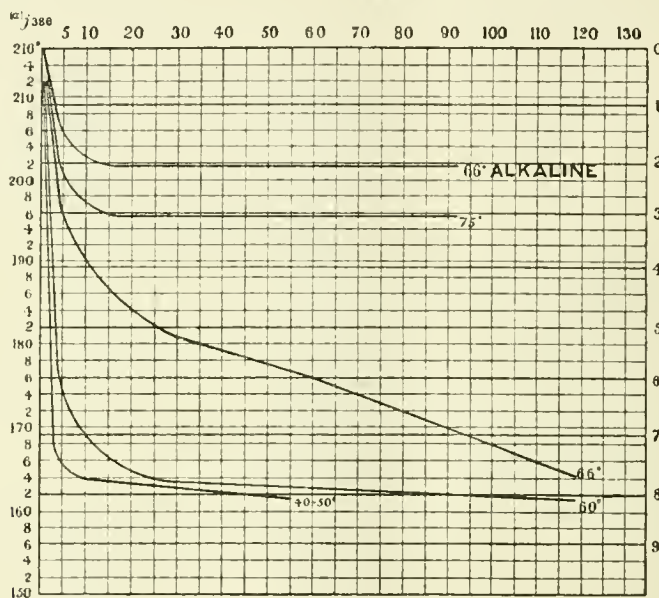
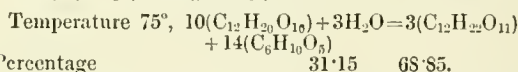


FIG. 1.

They give no formula for the reactions at the higher temperatures, but the re-action at 75° (167° F.) which seems to be tolerably constant, would be expressed by some such formula as this :—



The dextrin produced in this case is an achroodextrin, giving a colourless reaction with iodine. If the temperature of the reaction be higher than 75°, or if the mixture be rendered slightly alkaline, then the quantity of maltose produced is still less, and the dextrin is an erythrodextrin, giving a red reaction with iodine.

It will be noticed that in the presence of much malt extract the re-action at 66° (150° F.) after two hours comes very near to the normal. Brown and Heron suppose that starch consists of a compound molecule  $10(\text{C}_{12}\text{H}_{20}\text{O}_{10})$  and that, as hydration proceeds under the influence the diastase, one molecule after another splits off in the form of maltose, leaving dextrans of less complicated composition. (I have a model here to illustrate this.†) As the action proceeds

The results obtained by Brown and Heron differ considerably from those of O'Sullivan, though both point in the same direction. Both adopt 150° as the specific rotatory power of maltose, but while O'Sullivan states that the cupric oxide reducing power of maltose is 65 as compared with glucose 100, Brown and Heron give it at only 61. Such a difference will considerably affect the analytical results. As it turns out maltose may be made to reduce either the one quantity or the other, according to circumstances. Soxhlet\* has recently pointed out that the quantity of copper reduced is not a fixed quantity, either for maltose or glucose. It depends on the concentration of the solutions employed, and curiously enough the dilution of the solution has the effect of diminishing the reducing power of glucose, and increasing that of maltose. Accordingly the reducing power of the one substance cannot be expressed in terms of the other; indeed there is no reason for doing so. When a one per cent. solution of maltose is added to undiluted Fehling solution in excess, and the mixture is boiled for four minutes, an equivalent of maltose separates 6.1 equivalents of copper suboxide. If the Fehling solution is diluted four times and is not in excess, the reducing power rises to 6.4, but if the

arrangement, the whole of the remaining nine blocks suddenly changed to a red colour indicating erythrodextrin. The top remaining block was then hydrated, and removed changing to maltose, and leaving behind the second erythrodextrin. The third block was in like manner hydrated, and on its removal the remaining seven blocks changed to white, indicating the first achroodextrin. These were successively hydrated shewing the stages in the conversion of the starch molecules.

\* Journ. f. Prakt. Chemie., 1880, 21, 227.

\* Journ. Chem. Soc. (1879), II., 599

† A model was exhibited consisting of 10 superposed blocks each bearing the formula  $\text{C}_{12}\text{H}_{20}\text{O}_{10}$ , the formula being printed on a blue ground to indicate the iodine reaction. A molecule of water  $\text{H}_2\text{O}$  was attached to the top block, to illustrate the first hydration of the starch molecule. The upper block was then removed, with its attached molecule of water, representing a molecule of maltose, the formula of which  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$  was on a white ground. At the same moment, by a mechanical

Fehling solution is in excess it attains 6.53. Two other sources of error are pointed out by Soxhlet, viz., that excess of Fehling solution permanently blurs the paper of the filter owing to some compound of cellulose and copper being formed, and this, when the paper is burnt, will increase the amount of copper, and secondly that the determination of copper as oxide is not exact. To meet these difficulties he uses as a filter some asbestos which half fills the bulb of a chloride of calcium tube; on this the cuprous oxide is collected, and is then reduced to the metallic state by a stream of hydrogen. This reduction takes place at a very low temperature. According to Meissl\* the determination of the maltose by the polarising saccharometer is liable to error owing to the fact that maltose exhibits the phenomenon of birotation. A freshly-made solution being about 18° weaker than one which has stood some hours.

My own experiments confirm the statement of Brown and Heron as far as their reaction (No. 8) is concerned, viz., that at 60° (140° F.), indeed, 63° (145°), and below, gelatinised starch undergoes, in the presence of malt extract, a definite process of hydration, which results in the production of a saccharine matter, consisting for all practical purposes of maltose, 80 per cent, dextrin, 20 per cent. I have not succeeded in obtaining any trustworthy evidence of definite reactions representing the other numbers. I could never obtain results sufficiently concordant to warrant the assumption that at any given temperature the reaction can always be represented by a definite formula. All that is certain is, that when the reaction takes place at temperatures above 60° (140° F.), the quantity of maltose falls off, and the quantity of dextrin increases, until at 80° (176° F.), or thereabouts, the diastatic action is destroyed. I have endeavoured to put this in a graphic form on the diagram representing a thermometer. As you see the proportion of maltose and dextrin remains the same until 60° (140° F.) is reached. Even at 63° (145° F.) there is no practical difference; but from this point upwards the dextrin increases, until at 76° (169° F.) the proportion of maltose and dextrin are reversed. Up to this point the dextrin is one which gives no reaction with iodine. Above 76° (169° F.) the quantity of maltose produced is very small, and the dextrin gives a red colouration with iodine. At 80° (176° F.) the action ceases altogether, and iodine gives a blue colouration with the unaltered starch. What I wish you to observe is that the falling off in maltose is continuous, and does not advance by steps. The normal reaction by which 80 per cent. maltose and 20 per cent. dextrin is produced takes place, when sufficient malt is present, in about 20 minutes. After this reaction is complete the dextrin, in the presence of malt extract, undergoes hydration, and is ultimately converted into maltose; but this action is extremely slow, requiring a great many hours, and even then I have never seen a case in which the dextrin has completely disappeared. Here, again, the action is continuous. There is not the least indication of a halt corresponding to Brown and Heron's reaction No. 9. I think I am warranted in saying that there is no definite reaction, beyond the normal one, which will serve as a safe guide to the constitution of the starch molecule;

but as in the normal reaction, four-fifths of the whole is converted into maltose, it is evident that the formula,  $C_{12}H_{20}O_{10}$  must be multiplied by 5 at least.

The most favourable condition for the action of malt extract on starch is a slight acid reaction (malt extract is always slightly acid). Brown and Heron have pointed out that a slightly alkaline reaction produces much the same result as a high temperature. I find that an increase of acidity beyond a certain limited amount has precisely the same effect. (I used for these experiments lactic acid, as that is the acid chiefly present in malt, and is also that chiefly produced during fermentation.) It will be seen later on that this has a most important bearing on the fermentation of distillers' worts.

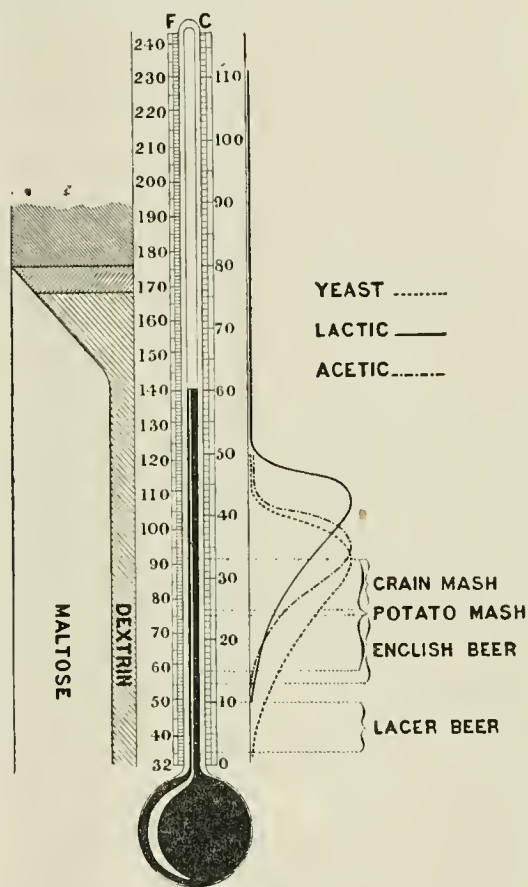


FIG. 2.

The body which brings about this remarkable action has received the name of diastase, but its nature and composition are very imperfectly understood, and it is doubtful whether it has ever been isolated. It occurs widely distributed in the vegetable kingdom. It is found most largely in germinating seeds, but also to a certain extent in seeds which have not germinated,\* in the buds of many plants, and in the leaves of the oak and wild thorn.† It sometimes occurs where there is no starch to convert, just as emulsin occurs in sweet almonds which contain no amygdalin. It belongs to

\* *Journ. f. Prakt. Chemie.*, 1881, 25, 114.

† Mr. Southby (Brewing Practically and Scientifically Considered, by E. R. Southby, London, office of "The County Brewers' Gazette, 1877.) after speaking of mashing temperatures of 148° to 153° F., says "With porter and stout it is a common practice in many large breweries to use a much lower temperature in the mash, and where great fulness is required, this lower temperature is an advantage, increasing as it does the proportion of the dextrin to the sugar produced from the malt." This statement is precisely the reverse of the fact, and in direct contradiction to all that we know of the influence of temperature on diastatic action.

\* In some parts of Russia, unsuited to the production of barley, but producing rye of excellent quality, this latter grain is treated in the mash tun without malt; the rye, without any preparation whatever, containing sufficient diastase to effect the conversion of its own starch. The produce of alcohol by this process is, however, from ten to fifteen per cent. less than when malt is used, and the method is not now so much employed as formerly.

† Adolf Mayer, *Lehre von den Chem. Fermenten*, 1882.



the class of enzymes, the soluble or unorganised ferments, the other principal members of which are ptyalin in the saliva, pepsin in the gastric juice, invertin in the wash waters of yeast, emulsin or sinaptase in the almond, papain in papaya carica, rennet, etc. They are without exception destroyed when in solution at a temperature below the boiling point of water. Diastase loses its properties at 80° (176° F.); invertin and pepsin about 50° (122° F.). In the dry state they are much more permanent. Diastase can be heated to 120° (248° F.), pepsin to 110° (230° F.), invertin to over 100° (212° F.). They all act by bringing about hydration, though under very different circumstances. Thus the ptyalin of the saliva, which is also capable of degrading the starch molecule, requires an alkaline reaction, which is, as we have seen, almost fatal to diastase. Pepsin requires a very decided acid reaction, and under these circumstances degrades the molecule of albumin, much as diastase degrades starch. The resulting peptones weigh more than the albumins from which they are derived, owing to the hydration which accompanies the reaction.

Diastase is not diffusible, and for that reason starch is unacted on in the cold, unless the external membranous envelope be broken by trituration with some hard body. I rather insist on this point, as there seems to be a good deal of misapprehension about it. Mr. Lovibond\* in his little book entitled "Brewing with Raw Grain," says at page 11: "Another method of forcing starch cells to give up their contents is to employ the osmotic or diffusive action of diastase. To accomplish this the ground grain is mixed with malt and water, and allowed to stand at as near 150° F. as possible, when the diastase of the malt finds its way through the cell walls, and converts the within contained starch into a solution of maltose and dextrin, which being very fluid readily escapes from the cells." This is quite a fancy picture of what does not, and could not happen. As long as the cell walls of the starch are intact no diastase can get in. But at 150° F. the cell walls are not intact. The diastase gets in at the crack, instead of diffusing through the membrane, that is all. I may mention another error based on a curious theory. A patent has been taken out by Mr. Kinder for rendering the action of the diastase on starch more complete by subjecting the mixture at 60° (140° F.) to a pneumatic pressure of about 80 lb. on the inch, just as starch is more easily converted by boiling with dilute acids, if the operation is performed under pressure. This is quite true in the case of dilute acids, but the pressure, *as pressure*, does nothing. The pressure is necessary only to get a higher temperature, which is not wanted for diastase, so that the analogy is altogether false. The action of compressed air was very carefully investigated by Paul Bert,† who found that while it retarded the development of the organised ferments, and in some cases even killed them, it had no influence whatever on the unorganised ferments, such as diastase, pepsin, etc.

A small quantity of diastase is capable of effecting the conversion of very large quantities of starch, probably 10,000 times its weight as a minimum. There is reason to believe, contrary to the generally received opinion, that the diastase itself undergoes no change, a very small quantity seems to do as much work as a large one, provided enough time is given, and this would hardly be the case, if the diastase was destroyed in doing the work. If this be the case the action is purely catalytic, and affords some clue to its nature. It is possible that the action of the enzyme

or soluble ferments is purely mechanical. In fact, that it is due to molecular vibration which causes the break-up of the molecules on which these ferments act. When solutions of albumin are heated to high temperatures, under pressure, they are peptonised, and undergo the same change which is brought about at lower temperatures by the presence of pepsin. Starch heated under pressure with water is converted into soluble starch. If this heating be continued at a pressure of 4 or 5 atmospheres, say at 150° to 160° (302°-320° F.)—the conversion into maltose or glucose (it is not clear which) is almost complete. In these two cases a certain degree of heat produces the same result as the soluble ferments. Heat is nothing but molecular vibration, so that there is ground for supposing that the soluble ferments are bodies in such a state of molecular vibration, that they shake asunder, as it were, the fabric of the starch molecule, or of the albumin molecule as the case may be. Somewhat, though not altogether, comparable to the shaking asunder of dynamite, or gun-cotton, by the detonation of a small quantity of fulminate.

The action of the soluble or chemical ferments is specific. Diastase will not invert cane sugar. Invertin will not break up starch. The diastatic ferments are without action on salicin or amygdalin. It has been supposed that diastase exercises a peptonising action on the albuminous matters of grain, and this view is adopted by Mr. Faulkner in his recently-published book. This is certainly a mistake. It is well known that the peptonising action proceeds best in very acid media. In Germany, where no top yeast is to be had from the breweries, yeast, which the distiller requires to add to his wort, is artificially cultivated in very concentrated worts, containing as much peptones as possible. In order to effect this the worts are allowed to sour, until indeed they contain nearly two per cent. of lactic acid. In such a medium diastase is coagulated, and rendered inert, so that the peptonisation must be due to some other ferment, which acts, like pepsin, in an acid solution, and which has received the name of peptase, or vegetable pepsin.

That several bodies of the diastase class exist in malt can hardly be doubted. Those which are capable of most completely breaking up the starch molecule are those which are most easily coagulated by heat. When extract of malt is heated, say for 20 minutes to 75° (167° F.), it is still capable of acting on starch, but the action does not go far, the proportion of maltose produced is small. If the action is continued for a considerable time it does not make much difference. This effect is quite independent of the temperature at which the reaction takes place, which may be much lower than 75°. It depends solely on the temperature to which the malt extract has been heated. The kind of diastase which alone is able to carry the action further has been removed by the heat. If we bring it back again, which we can easily do, by introducing a small quantity of malt extract which has not been heated, a further action on the partially degraded starch molecule at once sets in, and the reaction is rapidly converted into the normal one, represented by maltose 80 and dextrin 20 per cent. As this reaction is of considerable practical importance, I shall try to make this evident to you by means of a beam of polarised light.

The experiment consisted in passing a parallel beam of light from the electric lamp through a large Nicol's prism A by which it was polarised. The beam then traversed a trough B provided with plate glass ends *bb*, and a thin plate glass removable partition C, so as to divide the trough into two unequal cells. The light next encountered a plate of quartz C made up of two semicircles *dd* fitted together, one of which consisted of dextro-rotary, and the other of lævo-rotary quartz;

\* Brewing with Raw Grain, by Thos. W. Lovibond, London, E. and F. N. Spon, 1883.

† *Compt rend.*, 1875, lxxx, 1579.

both plates having a thickness of 3.75mm. The light finally passed through a second Nicol's prism D, which served as an analyser, and a lense E, by means of which an image of the quartz plate C could be projected on a screen. Matters being thus arranged, the larger division of the trough was filled with a solution of the product of the action of malt extract at 70° (158° F.) on starch paste. The smaller division, which was 1-10th the size of the larger one, was filled with unheated malt extract. By rotating the Nicol's prism D on its axis, the double semi-circular image of the quartz plate was brought to an uniform greyish-blue colour (sensitive tint). On removing the glass partition in the trough, the two liquids were enabled to mix. In a short time a change of colour took place on the screen, the right-hand semi-circle became purple, inclining to blue, while the left-hand one became red, indicating a fall in the specific rotary power of the solution in the trough, owing to the conversion of products formed at the higher temperature into those characteristic of the lower temperature. The effect was to some extent obscured by the colour of the solution, which cannot be entirely removed.

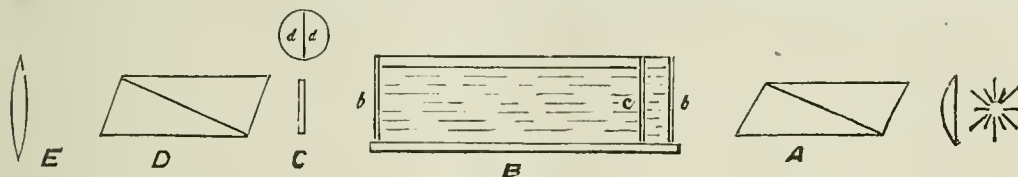


Fig. 3.

In brewing lager beer, hot water is not mixed directly with the malt, so as to produce at once, as in England, a mashing temperature of from 60° to 66° (140°-151° F.), but the malt is mixed with cold water. A portion of the mixture is run off, and is rapidly heated to the boiling point, to be returned to the mash-tun in order to raise the temperature of the whole. By this process the starch in the boiled portion is very completely utilised, but a considerable portion of it undergoes the saccharification corresponding to the higher temperatures, and ultimately the diastase is destroyed. This is of no consequence, for when the boiled portion is brought back to the cold malt and water in the mash-tun, the action is at once transformed into the normal one. The same thing happens in English distilleries where any starch not acted upon at the low temperature of the first mash, is transformed at the higher temperature of the first sparge into products corresponding with that temperature, and when this sparge is afterwards mixed with the first worts, containing unaltered diastase, the normal reaction ensues.

As we shall see presently, maltose is directly fermentable by yeast; dextrin is not. The distiller, whose object is to obtain the largest possible quantity of alcohol, is absolutely bound down to that mashing temperature which will give him the largest quantity of maltose. He may boil the rest of his grain to gelatinise the starch, but his malt must never go above the proper heat. Below the temperature at which barley starch gelatinises (57°-62°) he would lose the extract due to that starch, so he is obliged to work within very narrow limits. The brewer has more choice. Alcohol is not his only object, and he may vary the proportion of dextrin in his worts, and thus modify the character of his beer, by adjusting the temperature of his mashing. The distiller wishing to take advantage during his fermentations of the slow subsequent action of the diastase on the dextrin, must not exceed the temperature which would injure the diastase, and by boiling the worts destroys it.

## FERMENTATION.

The starch having been converted into soluble saccharine matter, the next step is to convert this more or less completely into alcohol. This is accomplished by means of yeast, which consists of minute globules or corpuscles endowed with life, and capable of reproduction. This is added to the wort, and fermentation is set up. If yeast were a perfectly pure substance, containing nothing but the cells of *Saccharomyces Cerevisiæ*, the matter would be a comparatively simple one, but it is not so. It is practically impossible to obtain yeast in a state of perfect purity. Other organisms are invariably present, each capable of setting up its own particular fermentation, and capable of reproduction at an alarming rate. Added to this, there are always minute organisms of various kinds adhering to the malt, and the different kinds of grain; and even the air which comes in contact with the worts is charged with the spores of all kinds of organisms, which are capable of development in the excellent conditions for their growth which all grain worts present. This is particularly the case with the air found in distilleries and breweries, especially if

the most scrupulous regard to cleanliness be not observed. Before we consider the conditions to be observed in order to obtain the maximum of alcoholic, and the minimum of secondary fermentations, we must study the peculiarities of the various organisms with which we have to deal. It would be quite impossible, in the time at my disposal, to deal with the immense number of tiny organisms which may, and occasionally do, produce the most disastrous results. I must confine myself on the present occasion to the three most important ones, viz., the yeast plant, the lactic acid ferment, and the acetic acid ferment. It was found impossible to project these minute bodies on the screen directly by means of the microscope, but with the assistance of my friend, Dr. Maddox, who is *facile princeps* in the art of microscopic photography, I have succeeded in preparing some photographs which I will endeavour to project. The yeast, as you see (autotype reproduction of photographs), consists of single cells of an ovoid shape. They are filled with matter of a highly nitrogenous composition, and in this transparent matter you will notice there are minute bodies, usually two or three in each cell, which probably consist of drops of liquid bedded in the protoplasm. In the healthy state, the walls of the cell are very thin, and scarcely visible, but when they are thick the cells are dead, or at any rate in a moribund condition. The cell is propagated in two ways. Under certain circumstances when the cells are growing in contact with air, spores are formed in the interior of the cell. The envelope then bursts, and the spores are set free; but the usual method of propagation during fermentation is by budding. A small protuberance is seen on the surface of the cell; this gradually increases in size, until it becomes as large as the parent cell. The new organism is finally detached, and then leads a separate existence; but before this takes place the new cell often becomes a parent in its turn, and this may go on until a perfect colony of cells hanging together is produced. There are two varieties of *Saccharomyces Cerevisiæ*. One



known as "top yeast," characteristic of distillers' fermentations, and those fermentations which take place in English breweries. The other, "bottom yeast," characteristic of the lager-beer manufacture. Though these two varieties may have had one source originally, they are not readily convertible one into the other, and are probably instances of the Darwinian hypothesis, the organism having been modified in the course of many generations by the conditions under which it has been forced to exist. (Both these were shewn on the screen.) For a sample of bottom yeast I am indebted to the Austro-Bavarian Brewery Company. There is another species of *Saccharomyces*, *Saccharomyces Mycoderma*, which, like the others, is propagated by gemmation. The peculiarity of this organism is that it forms a skin on the surface of worts after the alcoholic fermentation is complete. It is very frequently found on distillers' wash, and when floating on the surface it absorbs oxygen and produces acetic acid. When plunged below the surface it produces alcohol, but only in very dilute solutions. Natural yeast, which I may call "wild yeast," as it is not cultivated, is found in the fermenting juice of the grape, and some other kinds of fruit. It has received the name of *Saccharomyces Ellipsoideus*, from its shape. A specimen of this (the *apiculatus* variety), obtained from plum juice, was exhibited.

The lactic ferment is a very different organism, and belongs to the bacteria class. It consists of fine threads of variable length made up of rod-like cells united together. These threads are excessively minute, generally less than the  $\frac{1}{100}$  of a millimetre in diameter, though of variable length. This organism is fissiparous, that is to say, it is propagated not by budding, but by the division of the cells into two smaller cells, each of which leads a separate existence, and becomes as large as the parent cell, when it divides again. The acetic ferment consists of minute spherical cells which have a tendency to unite into threads, forming the well-known vinegar plant, which consists of millions of these bodies. These organisms are also fissiparous. All these bodies multiply under favourable circumstances with great rapidity, but in order to build up the substance of which they are composed they require certain kinds of food. These are carbohydrates, such as maltose or glucose, certain salts, chiefly phosphate of potassium and magnesium, and lastly albuminous, or rather albumenoid substances, which must be in the state of peptones, the molecule of albumin being too large to pass through the pores of the membrane which constitutes the envelope of the organism. In fact, they are unable to assimilate any food which is not diffusible. I have here some solution of pure invert sugar, divided into two portions. To both I have added a small quantity of yeast, and some phosphate of potassium, but to the one I have added some white of egg without any preparation, while to the other I have added a similar quantity of white of egg which has been previously treated with pepsin, and a little lactic acid. You will see that the yeast in the former has made no progress, while in the latter it has developed, and the solution is now in active fermentation. In other respects, the conditions of growth of these bodies present marked differences. By the side of the thermometer I have represented the effect of temperature. (See thermometer diagram, Fig. 2.)

The regularly dotted line represents the yeast curve. This organism is as you see capable of inducing a slight fermentation at a temperature very little over the melting point of ice. As the temperature rises its activity increases, until the optimum is reached at about 33° (92° F.) when it diminished down to nothing again, and at 50° (122° F.) or thereabouts it

is killed. The activity of the acetic ferment represented by the irregularly dotted line begins about 12° (54° F.) reaches its optimum about 36° (97° F.) when it begins to fall off, and finally, like yeast, loses its vitality at 50° (122° F.) The lactic ferment represented by the even line, begins to be active at a slightly lower temperature, viz., 10° (50° F.). Its activity increases gradually with the temperature, until it reaches a maximum at 44° or 45° (112° F.) when, as in the other cases, the activity diminishes, but observe how the line of life runs up, for although this organism produces no lactic acid at a temperature above 50° (122° F.), its vitality resists even the temperature of boiling water, and it is not finally destroyed under 111° (232° F.).

These curves are intended to indicate only in an approximate way the effect of temperature, and, temperatures being equal, they are very considerably modified by other conditions, for example, acidity. A certain acid reaction is favourable to the growth of yeast, and as lactic acid is the principal acid generally present in fermenting worts, its action may be taken as typical. In the presence of this acid, to an extent not exceeding  $\frac{1}{4}$  to 1 per cent., the yeast plant develops far more rapidly than in a neutral medium. On the other hand the lactic acid ferment is extremely sensitive to the product of its own action. In a solution containing one per cent. of lactic acid, this ferment develops with extreme difficulty, and when the acidity amounts to two per cent. or even a little less, the action is completely arrested, and the organism dies. For this reason the latter ferment is very rarely found in fermenting grape juice, on account of its great acidity. This organism grows best in a neutral solution, so that if we wish to prepare, by means of this ferment, large quantities of lactic acid from a saccharine solution it is necessary to add some substance, such as carbonate of lime, capable of neutralizing the acid as fast as it is formed. It sometimes happens in breweries and distilleries that, owing to slow cooling of the worts, they have remained too long at a temperature of about 40° to 45° (104° to 113° F.) which is especially favourable for the development of this ferment. In ordinary parlance, the worts have soured. It is a frequent practice to add whitening or some such substance to neutralise them. This is the worst thing that can be done. The ferment is not thereby destroyed, on the contrary it sets to work again with renewed energy. Acidity is favourable to the development of those organisms, which are known collectively under the name of mouldiness. If a mixture of yeast, bacteria, and mould be introduced into a neutral wort, the bacteria will gain the upper hand. If the acidity amount to one per cent. the yeast will predominate, and if the solution contain five per cent. of organic acids, the mouldiness will have pretty much its own way. It is a well-known fact that meat rapidly putrefies when subjected to the action of saliva, while it keeps a long time in gastric juice. The explanation is that the slight alkalinity of the former favours the growth of bacteria, while the acidity of the latter has the contrary effect.

As the lactic acid ferment is retarded and ultimately killed by the product of its own action, so also is yeast. When the liquid contains from 12 to 14 per cent. of alcohol, fermentation altogether ceases, and by still greater concentration the yeast plant is killed. Alcohol also exercises an unfavourable influence on all organised ferments, and it is chiefly to this circumstance, though not altogether, that fermented vegetable juices and worts, such as beer, wine, &c., and pharmaceutical tinctures owe their keeping properties. The action of the lactic ferment is very distinctly retarded by the presence of alcohol. The acetic ferment is not so sensitive. Many other



substances are antagonistic to the growth of these organisms. Benzoic, cinnamic, carbolic,\* salicylic, boric, and sulphurous acids, mercuric chloride, and generally the salts of the heavy metals, and in a less degree chlorides of the alkalis and alkaline earths. Hence the preservative action of common salt. Oil of hops and the aqueous extract of hops have a similar effect, and are powerful factors in the keeping properties of the highly-hopped English beers. The acids of the fatty series also retard fermentation, and this action appears to increase as we ascend the scale, acetic acid having a very marked influence in this direction, while butyric, valeric, and caproic acids, are powerful poisons for the yeast plant, and ferments generally. Our pickles would no doubt keep much better if prepared with valerianic or butyric acids, though the flavour might possibly suffer to some extent. On the other hand prussic acid and the poisonous alkaloids appear to have very little action.

Lastly, the various ferments interfere with each others' action. Even among these low organisms there is a struggle for existence, which does not always end in the survival of the fittest, or at least the fittest for our purpose. In a wort in active alcoholic fermentation containing abundance of healthy yeast growth the lactic ferment, for instance, develops much less slowly than it would under similar circumstances, in the absence of the yeast.

As a general rule yeast is less sensitive than the lactic and other organisms of the bacteria class to the influences I have named, particularly such as salicylic acid, hops, etc., so that the curves which I have laid down as approximately representing the action of yeast and lactic and acetic ferments, are very considerably modified according to the substances present in the medium in which they live and have their being.

Disregarding for the moment the other influences let us examine that of temperature. On the right-hand side of the thermometer diagram, I have indicated the range of temperatures incident to the two great systems of beer brewing, lager or bottom fermentation beer, and English or top fermentation beer. You will notice that within the lager beer range the lactic ferment, and the acetic ferment are for all practical purposes inactive, and the fermentation remains of necessity a purely or very nearly a purely alcoholic one, though, owing to the low temperature, it is very slow.

In the English beer range the conditions are altogether different. Although the action of the yeast is more energetic, we have got within the range of the other ferments, though their activity at this temperature is not great as compared with that of the yeast. Still, supposing the lactic ferment to be present in any quantity, the amount of lactic acid produced, as indicated by the curve, would result in a very sour beer. In practice this effect is considerably modified. Owing to the energy of the alcoholic ferment the lactic fermentation is to a great extent suppressed, and the abscissæ of the lactic curve are from this cause alone considerably shortened. In addition to this, the much larger quantity of hops used in English breweries has a tendency to lower both the curves, but it affects the lactic curve more than it affects the alcoholic one, and still further changes the aspect of the diagram. Still, as a net result, the English beer contains more lactic acid than the German beers. Professor Graham, in a paper on the brewing of lager beer, which he read before the first anniversary meeting of this society, said "that hops are to the English beer what ice is to lager beer—a means of keeping out the secondary ferments, and so promoting the preservation of the

beer." With this I quite agree, but with a single reservation, which is this. In the manufacture of lager beer, from the nature of the operation, the ice *must* be used, while in English beer, not of the high-class bitter beers, the hops are occasionally a little deficient. This is particularly the case with what are known as running beers, so called, I fancy, because you must run and drink them as soon as they are brewed.

Let us turn now to the influence of temperature on distillers' worts. In this case the object of the manufacturer is to convert the whole of the extract, or at any rate as much as possible, into alcohol, a thing not at all contemplated by the manufacturer of beer. The maltose being directly fermentable by the action of yeast, while the dextrin is not, the distiller must in the first instance so regulate his temperature as to obtain the maximum of the former with the minimum of the latter. He is therefore tied down to a mashing temperature not exceeding 60° to 63° (140°-146° F.). Up to this temperature he must go to ensure, as far as possible, the gelatinisation of the starch. When its conversion is complete, he may go a little higher, but not much. He is generally working (unless he is really a manufacturer of pure malt whisky) with as much raw grain as possible, and the minimum of malt, and to him a further action of diastase at a later period is absolutely essential. He dare not, therefore, play any tricks with it. This diastase, by long-continued action on the residual dextrin, is capable of gradually hydrating it, and converting it into maltose, but the action is excessively slow. It is found in practice that, in fermenting worts whose temperature has never exceeded 60° to 64° (140°-147° F.), that the dextrin is tolerably completely converted into alcohol, and it was assumed that the diastase acquired a greater power of acting on the dextrin when the maltose present in the worts had been removed by fermentation. In other words, that the maltose present in solution exerted a retarding influence on the action of the diastase. Closer investigation makes this view very improbable. The fact appears to be that, though yeast by itself has little or no action on dextrin, and diastase by itself only a very slow one, in the presence of both of these agents the dextrin is fermentable, though much more slowly than maltose or glucose. As I have already pointed out, diastase requires for its efficient action a slightly acid reaction; but when the acidity passes a certain point, the diastase is coagulated and rendered inert, so that if the worts during fermentation become very acid, the action of the diastase in assisting the yeast to ferment the dextrin becomes seriously impeded, and may be altogether stopped.

In order to obtain the maximum effect of the yeast, it is necessary to work at higher temperatures than in the case of beer, and the distiller has found that the best results are obtained when the temperature during fermentation rises to about 33° or 34° (92°-94° F.), which, by reference to the thermometer diagram, you will see is about the optimum of the alcoholic fermentation. But this temperature—indeed, the whole range of the distillers' fermentation, as practised in England—although extremely favourable to the yeast growth, is also extremely favourable for the lactic fermentation, and the acidity may grow to such an extent as to interfere seriously with the subsequent action of the diastase. Here there can be no question of hops or of ice to remedy the evil. Of course the thing is not so bad as it looks on the diagram, on account of the preponderating influence of the yeast, which, owing to the fact that one fermentation suppresses another, keeps the lactic ferment to a certain extent in check. It is on an active healthy growth of yeast that the distiller must

\* Benzoësaure, Carbonsäure, Salicylsäure, Zimmtsäure. Dr. H. Fleck, München, Oldenbourg, 1875.



chiefly rely to keep the lactic ferment down, added to which extreme cleanliness, and care in the selection of yeast, as free as possible from lactic ferment, to start his backs. Notwithstanding all precautions, a considerable quantity of lactic acid is unavoidably produced, and as this is produced at the expense of the maltose, which would otherwise be converted into alcohol, a very notable loss ensues. Every week some hundreds, I should say two or three hundred tons, of pure lactic acid is run to waste in the spent mash, from which alcohol has been distilled, in the United Kingdom alone.

As long as the lactic acid present is under one-half per cent., not much harm is done to the diastase; but as soon as this limit is passed, the diastase is very seriously lamed, and as a result a considerable portion of the dextrin remains unfermented, causing much loss to the manufacturer.

In Germany, where alcohol is largely manufactured from potatoes, the conditions are somewhat different. The distiller there is not troubled with an excise attenuation charge, and is consequently not obliged to separate the solid matter from the wort. He ferments the entire mash, solid matter and all. Under these circumstances he is able to prepare a much more concentrated liquor. While the worts in English distilleries rarely exceed a gravity of 1050, and more commonly are under 1040, the German distiller prepares a mash of a gravity of 1080 to 1090. This enables him to begin his fermentation at a much lower temperature, for the heat evolved in the fermentation of such a concentrated wort ultimately carries the temperature to the same point as reached by the English distiller, viz., the yeast optimum. Owing to the low temperature, the fermentation is at first rather sluggish, and it is only when the higher temperatures are reached that violent fermentation sets in.

When speaking of the influence of various substances on fermentation, I pointed out that the presence of alcohol had a restraining effect, but that alcohol affected the ferments of the bacteria class, such as the lactic acid ferment, far more than yeast. The German plan therefore presents the advantage that at the low initial temperature of the fermentation, comparatively little lactic acid is produced, and when the higher temperatures are reached, the mixture then contains so much alcohol that the lactic acid ferment grows with considerable difficulty.

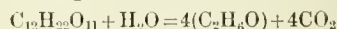
As long as the excise attenuation charge, which necessitates more or less clear worts, is imposed in England, this plan cannot be adopted here.

As the yeast rises to the surface of the fermenting wort, it is removed by the brewer, who obtains in this way much more than enough to start his new fermentations. The distiller cannot do this. As a general rule, distillers' worts, composed of the possible maximum of raw grain and the possible minimum of malt, contain such a small quantity of peptones, that in the later stages of the fermentation a portion of the yeast dies, and another portion lives on the substances, which pass into solution in consequence. As a result, the yeast contains a large quantity of dead cells. Even when the peptones are present in sufficient quantity, the lactic acid ferment has—especially at the end of fermentation, when the temperature is highest—developed to such an extent as to constitute a notable portion of the yeast collected. If this yeast were used to start the new backs, the result would be a great production of acidity, and consequent restraining of the action of diastase on the dextrin. In technical language the backs would "stick," and would refuse to attenuate beyond a certain point.

In this country the distiller, profiting by the excess

of yeast obtained by the brewer, draws his supply from that source, brewers' yeast being far freer from the lactic ferment than any the distiller can produce by the ordinary methods of working.

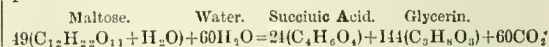
As to the nature of fermentation, more than one theory has been suggested. Pasteur observed that yeast, when growing in contact with air, and a proper supply of the salts, carbohydrates, and peptones, which constitute its natural food, rapidly absorbed oxygen, and under these circumstances produced comparatively little fermentation. He explained fermentation to be the result of the growth of the yeast cell, *in the absence of free oxygen*, a condition which compelled the cell to obtain its oxygen by the decomposition of the saccharine matter in the medium in which the cell is suspended. He pointed out that fermentation did not result in a simple splitting up of glucose (or maltose + water) into carbonic acid and alcohol, according to the formula



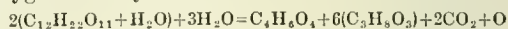
glycerin and succinic acid are always produced at the same time, and, according to Pasteur, 100 parts of glucose produce—

Alcohol .....	48.40
Carbonic acid .....	46.60
Glycerin .....	3.30
Succinic acid .....	0.61
Cellulose fat, etc.....	1.20

Supposing that the greater part of the sugar splits up into carbonic acid and alcohol, according to the simple formula, Pasteur explained the production of the succinic acid and glycerin by this very complicated formula.



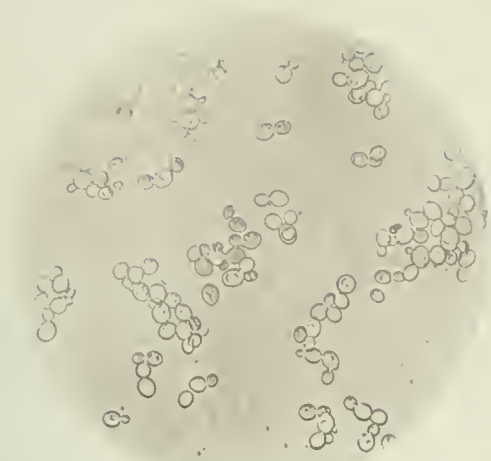
All this, however, does not explain precisely what is the source of the oxygen, in search of which the yeast is breaking up the saccharine matter.\* In order to meet this difficulty Monoyer\* constructed a far simpler formula, which explains the formation of the succinic acid and glycerin, and furnishes a supply of oxygen to the yeast cell.



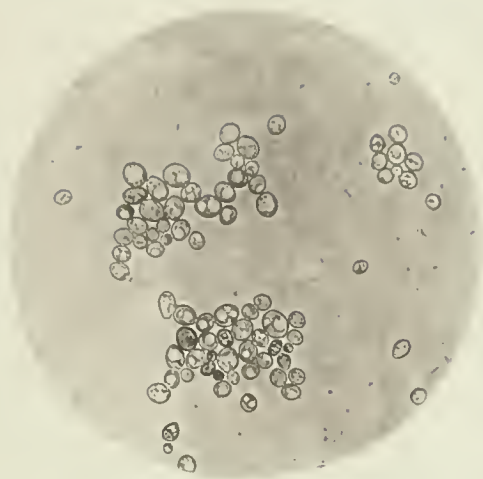
According to Nägeli† the action of the yeast cell is due to molecular vibration of the protoplasm which it contains. In consequence of this molecular action, the decomposition of the sugar takes place outside, and not inside, the cell, as Pasteur's theory requires. This action is supposed to extend to a minute but sensible distance from the cell. The formation of acetic ether by the combined action of the acetic and alcoholic ferments is certainly a fact which tells strongly in favour of this view. Acetic ether is produced when acetic acid and alcohol are brought together in the *nascent* state. Now, if acetic acid be formed in the inside of the acetic ferment cell, and the alcohol is produced in the inside of the yeast cell, no acetic ether could be produced; but if a zone of molecular vibration, in which the work is done, surrounds each cell, it is evident that wherever these zones cut each other acetic ether would be produced. This theory also explains—what is not readily explicable otherwise—the suppression of one ferment present in small quantity by another ferment present in large quantity. More powerful vibrations tend to suppress less powerful vibrations, which are not in unison with them, and if we suppose each ferment to have a peculiar vibration of its own, in virtue of which it sets up a specific decomposition in the surrounding medium,

\* Thèse de la faculté de Médecine de Strasbourg.

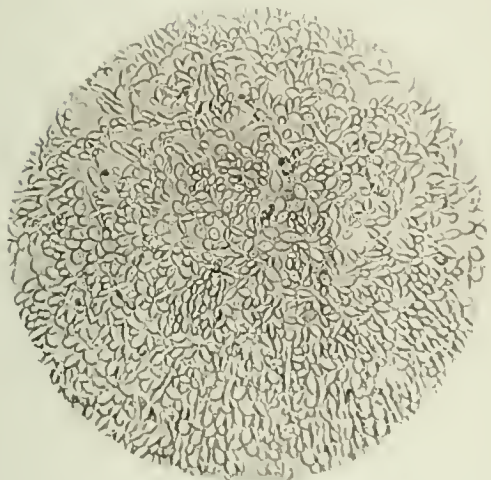
† Nägeli's *Gährungstheorie*.



*Saccharomyces Cerevisiae* (Var. Top Yeast)



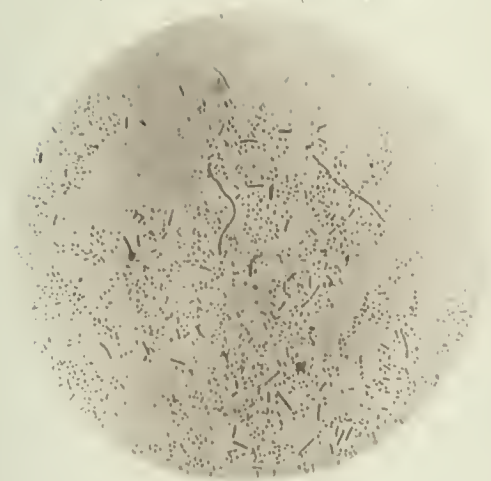
*Saccharomyces Cerevisiae* (Var. Bottom Yeast)



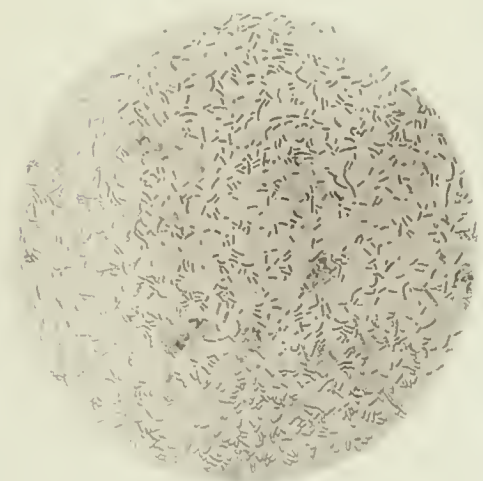
*Saccharomyces Ellipsoideus* (Var. *Aciculatus*)



*Saccharomyces Mycoderma*.



*Acetic Acid Ferment*



*Lactic Acid Ferment*.





we can easily understand how the predominating vibrations will tend to suppress the rest.

When speaking of diastase and the soluble ferments, I drew attention to the strong probability that their action is due to molecular vibration, and if Nügel's views about the action of the organised ferments be correct, we have a common explanation for the two phenomena.

I have endeavoured to sketch, as fully as the time at my disposal would permit, the processes concerned in the conversion of starch into alcohol, and the modern theories of the changes which occur. I am quite aware that this sketch has been a most imperfect one. Many important points I have only glanced at, others I have passed by altogether. I have confined myself pretty much to what takes place when matters proceed smoothly, without attempting to deal with the derangements caused by a host of organisms which from time to time intrude themselves. To the charge, therefore, of many and important omissions, I at once plead guilty, but I couple with that plea "extenuating circumstances."

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## Journal and Patent Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*A Hydrometer adapted to the Demonstration of Changes of Weight in Chemical Reactions.* By Max Gröger, Berl. Ber., 17,568.

THIS hydrometer is constructed on the plan of Nicholson's. The description of the instrument is accompanied by figures. It is so constructed that the sensitiveness is independent of the load, and depends only upon the diameter of the upper rod which carries the scale-pan: this being 1mm., the depression is 1cm. per 0.00786grm. The hydrometer is specially adapted to the lecture demonstrations described by Rosenfeld (Ibid. V., 14 and 16).—C. F. C.

*Pumps or Syphons for Drawing Acid from Carboys, &c.* J. Longshaw, Salford. Eng. Pat. 4873, October 13th, 1883. Provisional protection granted only.

THIS invention is a combination of a pump and syphon. A tube fitting into the mouth of the carboy or vessel is provided at its lower end with a ball or other valve. Its interior is fitted with a piston, the rod of which is hollow and bent over to form the longer leg of a syphon, its end being sometimes provided with a cock. The syphon is brought into action by drawing up the piston so as to fill the lower tube; on quickly pressing it down again the liquid passes up the bent piston rod and fills the tube, syphoning being thus established.—C. C. H.

*A New and Improved System of Evaporation and Distillation, and for Practical Application of the same.* W. A. Barlow, London. Communicated by P. Calliburee, Paris. Eng. Pat. 3309, February 14th, 1884.

THIS invention provides for the carrying into effect the above operations in a more economical manner than heretofore. The liquid operated upon is broken or pulverised into spray in the interior of a closed evaporating pan, and at the same time a current of air is forced through the spray so produced, which air has been previously purified and heated. The vapour carried off is condensed if desirable, and in processes of distillation the air discharged from the condensers is used through the apparatus again. The apparatus consists of: (1) A purifier, through which the induced current of air subsequently forced through the machine is filtered and washed. (2) A fan or other apparatus for forcing the air through the evaporator. (3) A heater, consisting of a vertical vessel provided with steam heated pipes, for heating the air to the desired point. (4) A pump for forcing the liquor under treatment through the pulveriser. (5) A domed evaporating pan containing the pulveriser, which consists of a cylindrical vessel provided with slits through which the liquid is forced, and striking against a hood breaks up the jets into spray. (6) The condenser, of which two or more may be employed in series, a pipe from the last of which communicates with the air-fan. The liquid in certain cases may be forced through the pulveriser by means of air, or a spray may be produced in a similar manner to an ordinary scent spray. Suitable apertures, provided with cocks, allow the material to be taken from the pan when required. The whole invention is covered by 12 principal and 9 minor claims, which include the combinations of the different apparatus, and also the evaporation of sugar, wine, beer, wort, grape juice, fruit syrups, milk, and the distillation of sea-water, alcohol, and petroleum, as well as the preparation of distilled water.—C. C. H.

*Improved Means for Generating Steam by Chemical Agents.* H. H. Lake, London. Communicated by H. Grüneberg and G. Hardt, Cologne. Eng. Pat. 4295, March 3rd, 1884.

THE generation of steam by means of chemical agents has usually been effected by the heat given out during



the formation of the hydrates of potassium and sodium from their oxides, caustic potash or soda, by the action of exhaust steam. The patentees in the present case state this method to be not only costly, but on account of the causticity of the materials, attended with danger to the operatives. In order to avoid this, burnt lime can be more advantageously used, the heat from which during slaking is used for the purpose stated. A double boiler is used, one containing water from which the steam is generated, the other the burnt lime. The slaked lime can be again converted into burnt lime and reused. The use of sulphuric acid, developing heat by its combination with water, is also claimed.—C. C. H.

*An Improved Titration Apparatus for Alkalimetric and Acidimetric Determinations.* C. D. Abel, London.

A communication from E. Greiner, Stützerbach, Thuringia. Eng. Pat. 4726, March 14th, 1884.

THE apparatus consists of a vessel charged with a standard solution, passing from which is a tube ending in a tap, the cock being formed by the lower end of a graduated burette. The whole is mounted on a wooden stand. On the one side is a bottle containing litmus solution or other indicator provided with a tube and compression tap leading to the titration vessel. The burette cock has both a vertical and two side passages. In order to charge the burette from the supply vessel, the cock is turned one quarter round to the right. To stop the supply, another quarter turn is made. To enable the liquid to run into the titration vessel, a further quarter turn is required, while a still further turn shuts off the standard, thus enabling the operator to perform a titration in a minimum space of time. The apparatus is very compact, and will prove an advantage where a large number of titrations are required in a small space of time.—G. H. B.

*Improvements in the Treatment of Asbestos, for Purifying the same and rendering it Non-corrosive in its Action upon Iron.* Eng. Pat. 4950, April 17th, 1884. John Dewrance.

CRUDE asbestos is known to have more or less corrosive action upon iron with which it may be in contact. This action is due to the presence of various soluble salts. The inventor removes these by a process of soaking in water and boiling in a dilute solution (1%) of caustic soda.—E. J. B.

## II.—FUEL, GAS AND LIGHT.

*Apparatus for Making Illuminating Gas.* Maurice Schwab, Manchester. Eng. Pat. 4550, Sept. 24, 1883. Being partly a communication from Jules Overhoff, Dortmund, Prussia.

THIS invention refers chiefly to a new form of generator, and an improved regenerative furnace for the production of lighting gas. The generator is situated centrally to the retort setting, and is fed through an opening in front, which is capable of being hermetically closed by a lid. The front wall is formed in steps, the blocks which serve for these being kept cool by the evaporation of water contained in trays, suitably carried by the walls of the generator. The grate consists of a central horizontal portion and inclined sides, whereby a large grate surface is obtained. The bottom of the generator slopes upwards towards the back, while the side walls are closer together at the back than at the front, and support a solid arch separating the generator from a chamber above, running the full length of the retort setting, and known as the distributing chamber. The gases from the generator enter this chamber by an aperture in the crown of the solid arch at the far end; and here the small particles of soot, etc., which have been carried over the sloping bottom of the generator, are deposited, and can easily be removed. The gases then pass out through apertures in the sides of the distributing chamber, mix with hot air brought in as presently described, and rise to the top of the setting, playing upon the retorts in their progress. They then descend on the outer side of these through the regenerator

to the main flue. The regenerator consists of passages on each side of the generator, built of a specially shaped brick, in such a way that an inner zigzag passage, serving as an outlet for the hot gases, is surrounded by a number of parallel passages, acting as inlets for the air. This latter is thus raised to a high temperature before it is allowed to mix with the generator gases emerging from the distributing chamber, as described above. Between the rows of retorts, vertical longitudinal walls are placed so as to divide the setting into four compartments—the two centres serving as up shafts, and the two outer as down shafts for the generator gases.—A. R. D.

*Improvements in the Production of Gas, Etc.* H. C. Bull. Eng. Pat. 4649, Sept. 29, 1883.

A CYLINDRICAL outer casing with a flat bottom and a dome top has its lower portion lined with firebrick and divided into radial sections by vertical firebrick walls, about 5ft. deep, each section being provided with a grate. This lower portion forms the producer, and has immediately above it a Körting's blower, the delivery pipe of which comes down through the firebrick walls and discharges under the producer grates. Above the blower is a vessel provided with tubes, through which the producer gases pass, generating steam from water contained in the tube-filled vessel. This latter is divided into two parts by a diaphragm, one part forming a steam generator, and the other a superheater. Outside the casing a number of cylindrical water vessels are situated, on the same level as, and each communicating with, the tube-filled vessel inside. The annular space between the steam generator and the outer casing forms a hot air chamber, whence a steam blast from the superheater draws a current through the Körting's blower, situated as above described. The producer gases, after passing upwards through the tubes in the steam generator, are conducted downwards through pipes surrounded by the air passages leading to the hot air chamber, and passing from the circumference of the casing to a hydraulic main, where the greater part of the tar and ammoniacal liquor separates out. The producer grates consist of a casting comprising a series of return tubular firebricks, leading from and returning to a front hollow bearer, and supported by a bearer at the back. By means of this arrangement water is heated for the supply of the steam generator above. The different sections of the producer are charged in rotation, so that the volatile portions of the fresh fuel, in one section, are driven off by the heat of the adjacent sections. Moreover, on account of the depth of fuel in the grates, which reduces the carbonic acid passing through it to carbonic oxide; and from the air and steam supplied by the injector being so highly heated as to require a minimum of air for the decomposition of the steam in the furnace, a gas is obtained, very low in nitrogen, but rich in combustible matters.—A. R. D.

*Gas Producers, Etc.* J. E. Bott, Manchester. Eng. Pat. 4681, Oct. 2, 1883. Complete specification not filed.

THE improvements herein described are mainly modifications in the mode of admitting the air to the interior of the furnace. The producer is, as usual, formed of a wrought iron casing, with a lining of refractory material, and the shape may be either circular, square or rectangular. A continuous tuyere, with an opening of a corrugated or wavy form, is placed in the interior of the furnace, along the sides in the rectangular form and circumferentially in the circular form; such tuyere being enclosed in a casing, into which the air or air and steam is forced by pipes from a heating box on the top of the producer. The heating box is supplied by means of a suitable blower and steam pipes. Regulating valves are used on the blast pipes to control the admission of air into the heating box. The results of thus introducing the air into the interior of the producer are, a uniformity of combustion, absence of cohering ash or clinkers, better control of the production of gas, and, being worked under pressure, no danger of combustion after leaving the producer, and no loss of heat in cooling tubes. Hoppers for the better admission of the fuel are also described, securing an uniform distribution.—C. C. H.

*Improvements in Burners for Burning Gas mixed with Hydrocarbon Vapour.* J. W. Sutton, London. Eng. Pat. 4747. Oct. 5, 1883.

FROM a metallic globe containing naphthalene, an outlet passes vertically downwards, and is connected with the centre of an Argand burner. Gas is passed into the globe by a three-way cock, and after mixing with the disengaged vapours of naphthalene, passes by the outlet to the burner. The flame of the Argand thus encasing its supply pipe prevents any condensation therein, and also heats the metallic globe above. A disc may be fixed below the globe to prevent it from being overheated. The three-way cock is used so that the gas may either be shut off altogether, or allowed to enter the body of the globe, or else may be admitted directly into the pipe supplying the burner. In this latter case there would be little or no admixture of naphthalene.—A. R. D.

*Generators for producing Gaseous Fuel.* J. Lones, C. Vernon, E. Holden, R. Bennett, Smethwick. Eng. Pat. 5353, November 13, 1883.

THIS invention provides for improvements in the general construction of gas producers, and mechanical methods for the removal of the ash and clinker formed by the fuel. A rectangular chamber is constructed of refractory brick and cast-iron, divided into two parts horizontally—the upper portion so formed being used as a combustion chamber, the lower as an ash pit, which is provided with openings for removing the débris, and smaller openings for the admission of a proper supply of air. The division of the two chambers is effected by a series of horizontal fluted rollers mounted in bearings, geared to each other

openings fitted with regulating valves for the admission of air, which can also be forced in by a steam jet, thus supplying a mixture of air and steam.

In order to remove the clinker formed in the combustion chamber, the fluted rollers are rotated, or a simple oscillating motion is given to them, and the ashes resting on and near them fall into the lower chamber. Clinkers or particles too large to pass between them are crushed during rotation, until they are small enough to fall through. A second modification is described, showing a construction of roller by building up in pieces forming openings for the admission of air instead of holes passing through the leaves of the rollers first described. The temporary interruptions for the removal of the débris as in ordinary producers are by the means described avoided. The patentees claim the construction of the generators as described, and the separation of the two chambers by means of the fluted rollers for the removal of the ash and clinker.—C. C. H.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Concerning the Occurrence of Diphenyl in Coal-tar oils.* K. E. Schulze. Berl. Ber. 17, 1203.

IN the year 1875 Fittig and Büchner (Berl. Ber. 8, 22) obtained from that portion of the coal-tar oils boiling at 242° and 243° C. a solid hydrocarbon having a melting point of 71°, and the boiling point of which was ascertained to be between 245° and 250°, and which on examination they found to be diphenyl. Later Reingruber (Ann. Chem. Pharm. 206, 167) mentions a body having

FIG 1

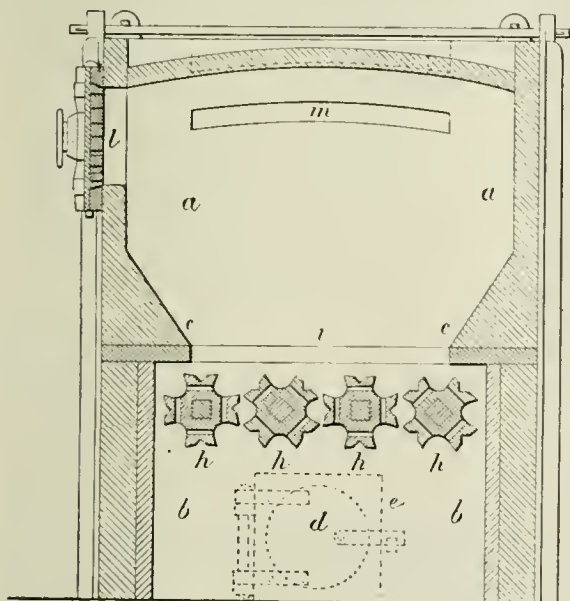
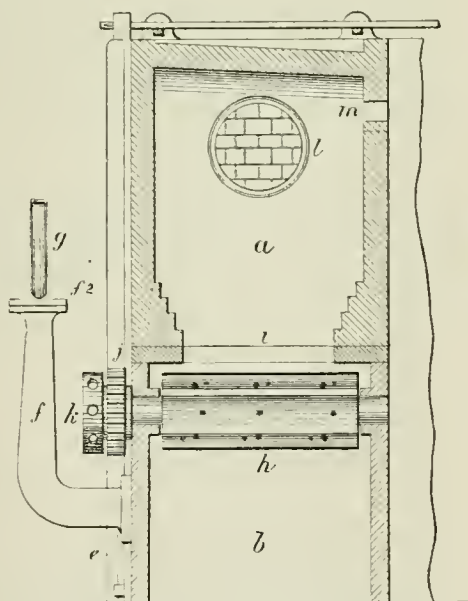


FIG 2



by spur wheels outside the producer, so that the whole are capable of complete or partial rotation on their axes, by means of a winch handle attached to one of their axes. These fluted rollers form the plate or bed of the furnace supporting the fuel, and have suitably constructed openings formed through them for the admission of air from the lower chamber to the burning fuel in the upper part of the producer. The grooves on the rollers are alternately deep and shallow, so arranged that in rotation the leaves or lobes of the one come nearly into contact with the corresponding parts of the other. The combustion chamber is provided with an opening for the admission of fuel, and a flue for conducting away the gaseous fuel. The lower chamber is provided with

the melting point 71°, but believed it to be impure naphthalene. The author by carefully fractionating that portion of the distillate boiling at 200° and 300°, obtained an oil coming over between 245° and 255° which solidified on cooling to -15°. After draining off the adhering liquid and expressing the residue, he found it consisted essentially of  $\beta$ -methylnaphthalene. On treatment with about 75%  $H_2SO_4$  at a temperature of 40° to 50° the product gave the characteristic smell of diphenyl, and by cooling, draining, pressing and recrystallisation from alcohol the compound was obtained pure. Later the author found that the portion coming over between 242° and 245° also contained diphenyl, and that by treatment with  $H_2SO_4$  it solidified, showing a



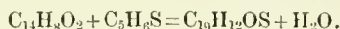
melting point  $68^{\circ}$ . Repeated re-crystallisation from alcohol gave pure diphenyl. The author is of opinion that the homologues of diphenyl are present in small quantity in tar, but has not yet obtained the  $\gamma$ -ditolyl melting at  $121^{\circ}$ , although an oil boiling at  $267^{\circ}$  (uncorr.) giving the characteristic smell of the diphenyl compounds has been obtained and is probably a methyl diphenyl.—G. H. B.

On  $\alpha$  and  $\beta$ -Methylnaphthalene. K. E. Schulze. Ber. 17, [6] 842.

BOTH these compounds occur in creosote-oil.  $\beta$ -Methylnaphthalene crystallises like naphthalene in large white plates, which melt at  $32^{\circ}5$ . It boils at  $241^{\circ}$  to  $242^{\circ}$ .  $\alpha$ -Methylnaphthalene is a liquid of pleasant aromatic smell; it boils at  $240^{\circ}$  to  $243^{\circ}$  and does not solidify at  $15^{\circ}$ . In all its properties it is exactly similar to the  $\alpha$ -methylnaphthalene prepared synthetically by Fittig.—D. E. J.

Laubenheimer's Reaction. Edgar Odenheimer. Ber. 17, [9] 1338.

LAUBENHEIMER has shown that Phenanthrenequinone can be detected by the deep blue colour which it produces with toluene and concentrated sulphuric acid, and according to V. Meyer, this reaction is due to the methyl-thiophene contained in crude toluene. The author has prepared and analysed the blue colouring matter and finds that it has the composition  $C_{20}H_{12}OS$ . Most probably it is produced, according to the following reaction, from the phenanthrenequinone and thiutoluene:—



It is a dark blue powder, insoluble in water, but easily soluble in alcohol, ether, benzene, &c. It is remarkable that this blue dyestuff, when oxidised with lead chromate, yields anthraquinone. Probably the methyl-group of the methyl-thiophene takes part in forming the anthraquinone, for the green dye which is obtained from thiophene and phenanthrenequinone does not yield anthraquinone when treated in the same way.—D. E. J.

The Acridines. August Bernthsen. Ann. Chem. 224, 1.

IN this paper the author gives a complete account of the recent researches on acridine and its derivatives. The reaction by which acridine is synthetically prepared from diphenylamine and formic acid has been shown to be a general one, and the author proposes to call the class of bodies thus prepared by the general name "acridines."

The acridines are distinguished by the following characteristics:—

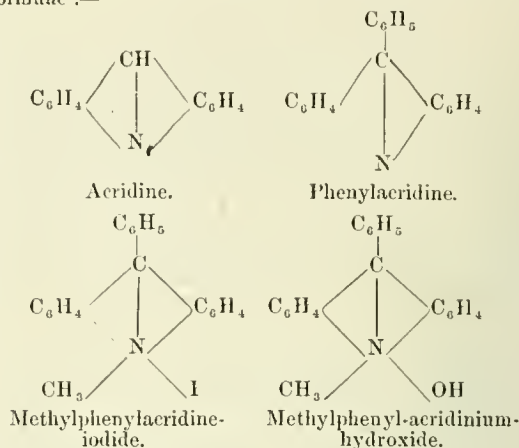
- (1) The fluorescence of the diluted solutions.
- (2) The formation of ammonium salts by combination with methyl iodide,  $CH_3I$ , &c.
- (3) And especially the formation of hydroacridines on reduction. These hydroacridines are *not basic*, and can easily be reconverted into the original bases.

Among the bodies belonging to this class, which have already been prepared, the most important are:—

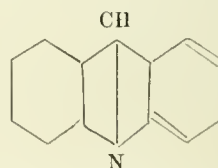
Acridine itself; from diphenylamine and formic acid.  
Methylacridine; from diphenylamine and acetic acid.  
Butylacridine; from diphenylamine and valeric acid.  
Phenylacridine; from diphenylamine and benzoic acid.  
Acridylbenzoic acid; from diphenylamine and phthalic acid.

Of these, phenylacridine has been most completely studied, and as an example of the "ammonium salts" formed by the acridines, we may mention methylphenylacridinium iodide,  $C_{19}H_{13}N + CH_3I$ , which is formed when phenylacridine is heated in closed tubes, together with an excess of methyl iodide. Caustic soda converts this into the corresponding ammonium base, viz., methylphenylacridinium-hydroxide,  $C_{19}H_{13}N, CH_3OH$ . In these compounds we must assume the presence of pentavalent

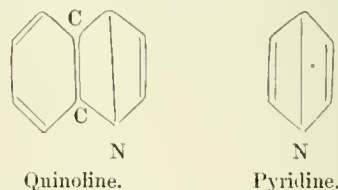
nitrogen. Their constitution and their relations to acridine, &c., are shown by the following structural formulae:—



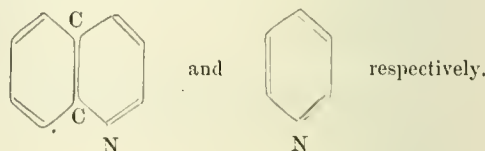
The above formula for acridine can be written thus:—



and it is probable that the researches of Graebe and Caro on the oxidation-products of acridine (acridinic acid, &c.), and their relation to the Quinoline and Pyridine derivatives, will lead to the adoption of the constitutional formula:—



instead of those now in use, viz.—



D. E. J.

An Improved Process for Calcining Bones, Peat, etc. Eng. Pat. 5064, Oct. 24, 1883. B. J. B. Mills, London. A Communication from A. Zwilling, Vienna.

A cast-iron cylinder, set in a non-conducting casing, is provided with an opening at the top for the admission of superheated steam, an outlet worm leading downwards, and a small outlet or test tube. This cylinder is filled with bones and then steam is gradually allowed to enter it through the superheater, the supply being regulated by a stop-cock. Steam is not turned on full until the worm has become hot and an ammoniacal smell proceeds from the test tube. The gaseous products after passing through the worm, where the greater part of the steam is condensed, are conducted through absorbing vessels where the ammonia is removed by dilute sulphuric acid, and the remaining small quantities of cyanides by solution of sulphate of iron. The remaining gas, freed from carbonic acid by passing through a lime purifier, is excellently

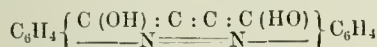
adapted for illuminating purposes. The steam is conveniently superheated by passing it through iron pipes kept at a low red heat, the steam gauge of the boiler meanwhile indicating a pressure of two atmospheres.

As a protection against the action of the fire, the iron pipes are coated several times with a mixture of two parts of clay and one part of fossil meal made up into a thin syrup with water. Spent bone black may be revived in the same way. This operation generally takes two hours-and-a-half; the calcination of bones requires five hours. If, when the process is completed, ordinary steam be driven through the cylinder for five minutes, the bone black may be taken out at once without previous cooling. To utilise all the heat of the superheated steam it is well to employ a series of cylinders so fitted with stop-cocks and other connections that each may in turn be made the first of the series, and then taken out of connection for emptying and recharging. This process is also applicable to the calcination of peat and other substances containing a rich percentage of carbon. Simplicity, cheapness, safety, and convenience, are claimed as the advantages of the invention.—A. R. D.

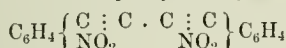
#### IV.—COLOURING MATTERS AND DYES.

*On the Constitution of Indigo-Blue.* P. Alexejew. Ber. 17 (Ref.) 172.

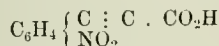
The author considers that the structural formula



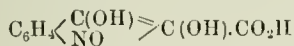
represents the constitution of indigo, and explains its production from dinitrodiphenyldiacetylene,



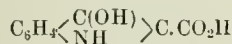
as well as from orthonitrophenylpropionic acid,



In the latter case the hydrate of isotogenic acid,



is first formed, and this is reduced to indoxyllic acid,



Like indoxyl, indoxyllic acid is easily converted into indigo-blue.—D. E. J.

*Chlorbenzaldehyde and Chlor-indigo.* R. Gnehm. Ber. 17. [6] 752.

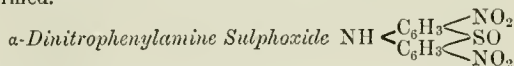
NONE of the methods yet employed for the artificial production of indigo have been commercially successful, one of the chief difficulties being the high price of orthonitrobenzaldehyde. When benzaldehyde is treated with nitric acid, it is converted chiefly into metanitrobenzaldehyde, only a small quantity of the orthonitro-compound being produced. The author has endeavoured to overcome this objection by employing, instead of benzaldehyde, a substituted benzaldehyde in which the ortho-position is unoccupied. He starts with dichlorbenzaldehyde  $\text{C}_7\text{H}_4\text{Cl}_2\text{O}$ , which is treated with an excess of nitric and sulphuric acids; on pouring into water, the nitro-product formed separates out, and can be crystallised from alcohol. This body is orthonitrodichlorbenzaldehyde, and by the action of acetone upon it in presence of caustic soda, chlor-indigo is obtained (just as orthonitrobenzaldehyde and acetone yield ordinary indigo). Tetrachlorindigo,  $\text{C}_{16}\text{H}_6\text{Cl}_4\text{N}_2\text{O}_2$ , is very similar to common indigo-blue both in appearance and properties. Reducing agents convert it into tetrachlorindigo-white; but the reduction does not proceed so easily as in the case of common indigo, and the indigo-

white produced is not so easily soluble in alkaline liquids. These peculiarities render the preparation and use of the dye-bath rather difficult; but in other respects the results are satisfactory.—D. E. J.

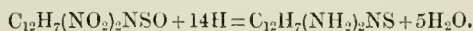
*Methylene Blue and allied Colouring Matters*, by Aug. Bernthsen. Berl. Ber. 17, 611.

THE author has further investigated the thiodiphenylamine, prepared by him by the action of sulphur upon diphenylamine. (Ibid. 16, 2896.)

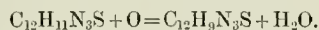
Treated with nitric acid (Sp. Gr. 1.44) at  $0^\circ\text{C}$ . it is easily nitrated, the sulphur being simultaneously oxidised, two isomeric Dinitrophenylamine-sulphoxides being formed.



is obtained as a yellow crystalline powder; it is but slightly soluble in alcohol and in acetic acid. When treated in solution in hydrochloric acid, with stannous chloride, it is reduced according to the equation :



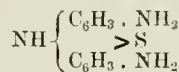
By nitrating the acetyl-derivative of thiodiphenylamine the corresponding  $\alpha$ -nitroacetyl-diphenylamine sulphoxide  $\text{C}_{12}\text{H}_6(\text{NO}_2)_2\text{NC}_2\text{H}_3\text{O}\text{SO}$  is obtained. The isomeric  $\beta$ -derivatives are more soluble than the  $\alpha$ -compounds above described; they are isolated from the mother-liquor from which the latter have separated. The derivatives obtained by reduction of the nitro compounds are the leuco-bases of violet colouring matters. The  $\alpha$ -derivative was converted by oxidation of the leuco-base into a body which was found to be in every respect identical with Lauth's violet. (Ibid. 12, 2069.) The oxidation to the violet takes place according to the equation :



The isomeric colouring matter obtained from the  $\beta$ -compound is of a much redder tone and further differs from the  $\alpha$ -violet in being dissolved by concentrated sulphuric acid to a violet coloured solution, the former dissolving to a green solution. The  $\beta$  hydrochloride is also considerably more soluble in water than the  $\alpha$ .

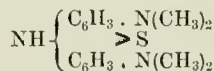
*Methylene Blue.*—The near relationship of this compound to the violet above described is evident from the comparison of the constitutional formulae of their leuco-bases :

*Diamido-thiodiphenylamine.*



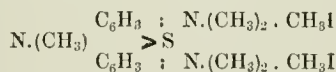
or Lauth's white. And

*Tetramethyldiamido-thiodiphenylamine.*



or Methylene white.

Both bases yield the same methylated derivative as the final product of the action of methyl iodide, viz., the dimethyliodide of the pentamethyldiamido-thiodiphenylamine—



With regard to the positions of the N-atoms in the benzene complex, the formation of these colouring matters respectively from paraphenyldiamine and para-amido-dimethyl-aniline, leaves no doubt that the N-atoms occupy to one another the 1 : 4 position.

The conversion of Lauth's violet into methylene blue, by methylation, is a question which is still under investigation.—C. F. C.



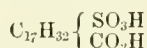
# V.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## *The Composition and Mode of action of "Turkey-red Oils."*

A Müller-Jacobs. Dingler, 251, 499-506. A previous paper of the author's "On the Theory of Turkey-red Dyeing" is to be found in *Soc. Indust. Mulhouse*, February, 1883. The first part of the present communication deals with the author's experiments, which in the second part are compared with those of other investigators.

WHEN any oil having the composition of a triglyceride is treated with strong sulphuric acid, so that the temperature of the mixture never exceeds 50°, and so that no evolution of sulphur dioxide takes place, and the mixture quickly diluted with double its volume of cold water, there is obtained either a clear solution or a milky emulsion, from which oil drops separate, according to the kind of oil taken, the duration of the action, etc. By dissolving the product of the reaction in double its volume of ether, and shaking with about ten times its volume of water, an aqueous and an ethereal solution are obtained. The aqueous solution, on treatment with hydrochloric acid or common salt, allows an oily layer to separate containing some 60 per cent. of water; this is for the most part removed by addition of common salt. The ethereal solution is heated to remove the ether, and the residue extracted with warm alcohol.

The substance soluble in water is strongly acid, easily soluble in acidified water, and miscible with ether; the solutions froth on shaking. The substance or its solution heated alone or with addition of acids or alkalis, or allowed to stand a long time, is decomposed with elimination of sulphuric acid and formation of a yellow crystalline substance, melting at 59-63°, to be afterwards described. With the alkalis salts are formed, syrupy masses soluble in alcohol and water and not decomposed at 100-110°. These salts of the alkalis, treated with the acetates of the heavy metals, yield a series of characteristic salts, of which the barium, copper, and silver salts have been analysed; the free acid was also analysed after drying over sulphuric acid. The numbers obtained, all point to the substance having the composition  $C_{18}H_{34}SO_5$ , i.e., sulpholeic acid:—



The composition of the acid is also shown by its products of decomposition. The purified acid was boiled for two hours with twenty times its volume of water, and the separated oils collected, m.p. = 62°·5; the aqueous solution was free from glycerine, but contained sulphuric acid, the amount of which was estimated. The results obtained show that the decomposition is expressed:—



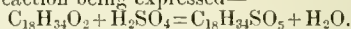
The separated oily product is completely soluble in ether and in alcohol, and from the alcoholic solution deposits a white crystalline substance, melting at 70°·4. This solid acid, and also its copper and barium salts, were analysed, the results confirming the above formula,  $C_{18}H_{36}O_3$ , i.e., oxystearic acid. After the separation of this acid from the alcoholic solution there remained dissolved a liquid acid, viscous and not crystallisable at -10°; the analysis of the lead salt shows that this substance is identical with the oxyoleic acid obtained by Burg and Overbeck. The free sulpholeic acid combines with two atoms of iodine or bromine; the free acid and also its salts soluble in water dissolve easily sulphur, iodoform, camphor, naphthalene, anthracene, colouring matters and lakes; they mix with ether, chloroform, carbon bisulphide, turpentine, benzene, petroleum, ethereal oils, etc.; and these mixtures either mix with or dissolve to clear solutions in water. Similarly, tolerably large quantities of triglycerides (oils), oleic acid, and solid fatty acids are dissolved; such a solution of a glyceride on boiling is decomposed, with liberation of glycerine, and if metallic salts are added they are carried down along with the liberated acids.

The substance soluble in ether is simply the triglyceride which has been unacted upon by the sulphuric acid.

The substance soluble in alcohol can be separated into a crystalline portion melting at 70°·5 and a liquid portion, and their properties are the same as those of oxyoleic and of oxystearic acid formed by the decomposition of sulpholeic acid as just described; hence during the action of the sulphuric acid a portion of the sulpho-acid at first formed is decomposed.

The acid solution from which the sulpholeic acid was separated by the addition of hydrochloric acid or of salt, contains glycerine.

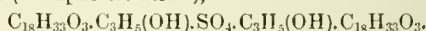
By the action of strong sulphuric acid on pure oleic acid a reaction takes place similar to that when a triglyceride is employed, i.e., a fair portion of the oleic acid is converted into sulpholeic acid and its products of decomposition, oxyoleic and oxystearic acids, the primary reaction being expressed—



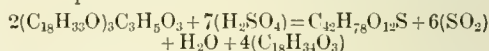
Ricinoleic acid is also similarly acted upon, but the reaction requires a much greater length of time; the sulpho-acid formed appears, however, to possess greater solvent powers for oils, etc., than the sulpholeic acid does.—H. B.

## *On the Composition and mode of Action of Turkey-red Oil.* By A. Müller-Jacobs. Dingler 251, Heft. 13.

Liechti and Suida (see this Journal vol. II., 540) believe that the above-mentioned compound, which is soluble in water, is a compound containing glycerine, consisting of the monosulphate of hydroxy-di-glyceryl oleate ("sulpholeic acid"),

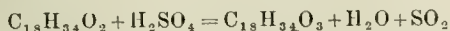


They lay stress on the fact that, during the reaction, in every case very considerable quantities of  $SO_2$  are evolved, and that they have not been able to avoid the formation of the same. Moreover they state that no glycerine at all is contained in the sulphuric lye. This statement is in direct opposition to the observations of Frémy and others. As it has been found that bad results are obtained with the product got simultaneously with a considerable evolution of  $SO_2$ , this is carefully avoided in the process of manufacturing Turkey-red oil. The author's mixtures, including those with oleic acid, are prepared with special reference to this point. In no case did an evolution of  $SO_2$  take place, and none could be found in the lye. A glance at Liechti and Suida's equation



must show to anyone acquainted with the manufacture of Turkey-red oil that it does not at all correspond to what actually takes place. Various manufacturers have attempted the regeneration of glycerine from the lyes, and not without success, but the quantities obtained did not pay even the expenses of production. Apart from this the incorrectness of Liechti and Suida's mono-sulphate formula is shown by the fact that the substance soluble in water can also be obtained to the extent of 50% from pure oleic acid, provided care be taken to avoid evolution of  $SO_2$  and consequent decomposition of the sulpho-acids. This was entirely left out of account in Liechti and Suida's experiments in the same direction, and only small quantities of a body "soluble in water" were obtained by them. Liechti and Suida, in basing their theory of the mode of action of Turkey-red oil on the decomposition products of the compound soluble in water, have entirely omitted to allow for the 30% of substances insoluble in alcohol and consisting chiefly of unchanged triglycerides, which are always obtained in actual practice. But it is specially to these compounds that the principal action in mordanting is due, otherwise a Turkey-red oil prepared from oleic acid should have the same mordanting power as that consisting of triglycerides, which is by no means the case. If the cotton to be dyed was mordanted with the ammonium salt of the pure compound soluble in water—sulpholeic acid—author in all cases obtained poor results on dyeing, but the colour was everything that could be desired when about 10% of any triglyceride was previously added to the sulpholeic acid. At the beginning of the paper in question, it is mentioned that Frémy, when studying the

action of oil of vitriol on olive oil, was unable completely to separate the products of the reaction, and a few lines further on great stress is laid on the empirical constitution found by him for the barium salt of the sulpholeic acid. Frémy cannot have worked with the pure sulpholeic acid, since this is not soluble in acidified water. Although Liechti and Suida have criticised my formula as per my patent for the preparation of solid oleic acid from oleic acids and glycerides of the same (see 1882 [244] 172), yet they have not repeated the experiment in the manner indicated by me, and consequently arrive at totally different conclusions which do not all correspond to facts. Their equation



gives absolutely no explanation whatever of the intermediate product soluble in water, which is well known to every calico printer; and indicates the presence of sulphuryl where none can be proved present. By one recrystallisation of the mass an acid was obtained of a much higher melting point than that given by Liechti and Suida for oxyoleic acid, viz.,  $58^\circ C$ , and this shows that a careful examination of the circumstances in question cannot have been made. With respect to these differences it seems probable that Liechti and Suida did not work with perfectly pure sulpholeic acid, but with a body containing in solution considerable quantities of unchanged glycerides and oxyacids. If it be attempted to investigate the mode of action of "Turkey-red oil" by means of analytical results, it is essential that the so called older Turkey-red processes should be compared more closely. S. Jenny (Soc. Ind. Mulhouse, 1869, pp. 10 and 335), after making a pretty extensive series of experiments, has already expressed the opinion that unchanged oil was present in the "Huile Tournante" emulsions, besides various soaps, and that this very unchanged oil surrounds the colour lake, in a manner keeping it damp and protecting it from exterior influences. When using Turkey-red oil, a single passage does not suffice to bring out an equally bright colour to that obtained by the old process, since the quantity of triglyceride fixed is too small, but the red obtained is quite perfect as far as solidity is concerned. The salts of sulpholeic acid which are not removed by washing likewise act as fixing agents, since they combine with alumina. The difference in mordanting action between the ammonium salt and the potassium or sodium salt of sulpholeic acid is caused by the different facility of decomposition of these salts. The salts of the fixed alkalis remain unchanged on drying the goods, and are washed out with the greater part of the unaltered oil; the ammonium compound on heating is decomposed into free ammonia and oxyacids, together with sulphuric acid, and, at the same time, the unaltered triglyceride is fixed in the goods; hence the conclusions which must be deduced are the following:—

The so-called Turkey-red oil is to be considered as a saturated solution of unchanged oil (triglyceride) with decomposition products of sulpholeic acid in the alkali compounds of the same, or some analogous acid such as sulphorcinoleic acid. Its action as a mordant is due to its yielding oil to the fibre in a finely divided form. In the best processes the sulpholeic salts are removed as much as possible by washing, but in simpler processes these are fixed with alumina.

The admitted better action of the ricinoleic acid mordant (due to the easy solubility of castor oil), is due to the fact that such a mordant contains more oil dissolved in the corresponding sulphuric acid, and hence yields more to the fibre than other triglycerides. The experiments of Runge, Mercer, Greenwood, and Keyser were fruitless, which is natural enough since in their processes the evolution of  $SO_2$  was inevitable. The so-called "acide sulpho-oleique" did, also, not gain ground owing to its being wanting in the main characteristics of "Turkey-red oil," viz., complete solubility in water with a definite amount of unchanged oil. The product obtained by converting triglycerides into emulsions by means of solutions of gelatin and hypochlorites of the alkalis does approach what is required, since it yields a genuine bright red with one passage. Cf. author's paper, 1873 [210] 236, and C. Romen's "Die Colorie der Baumwolle

mit besonderer Berücksichtigung der Türkischroth-Färberei," (Vienna, 1878, A. Hartleben). By closely following this and similar lines, author was at last able to manufacture that product which is at present still known in commerce as "Turkey-red oil." Author absolutely declines participation in the invention of that Turkey-red mordant during the preparation of which  $SO_2$  is evolved.—H. A. R.

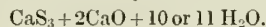
*Producing Printing Blocks by means of Photography.*  
Walter Bentley Woodbury. Eng. Pat. 4735, Oct. 5, 1883.

THIS invention has received provisional protection only. The process consists in breaking up the continuous half-tone of negatives produced in the ordinary way, into a series of lines or dots, by combining with the original negative a negative produced by photographing such objects as fine ruled lines, netting, gauze, dots, or a print from a grained stone. A similar effect may be produced by dusting a fine opaque powder over the image. Plates so prepared are transferred to metal, so as to form a printing block in any of the well-known ways.—C. F. C.

## VI.—ACIDS, ALKALIS, AND SALTS.

*On certain Compounds of Sulphur.* A. Geuther. Ann. Chem. 224, 178.

I. The oxysulphides of calcium. These differ from the other known oxysulphides (such as carbon oxysulphide and the oxysulphides of vanadium) inasmuch as they have no representatives among the typical calcium compounds; they are compounds of a polysulphide with a monoxide, and contain also water of crystallisation. Two such compounds are known; of these the first is prepared as follows:—50gm. of flowers of sulphur and 100gm. of slaked lime are boiled with 2 kilos. of water in a loosely-covered vessel for six hours. Evaporate down to one-quarter of the bulk, filter hot, and allow to stand for a week or fortnight in a well-corked flask in a cool place. The oxysulphide separates out in yellow prisms, which are freed from mother-liquor by pressing between filter-paper; they cannot be washed, for they decompose in contact with water, leaving a residue of calcium hydroxide. When treated with concentrated HCl scarcely any sulphuretted hydrogen is evolved, but almost colourless drops of hydrogen persulphide  $H_2S_3$  (A. W. Hofmann) are produced. The author considers that this indicates the presence of a trisulphide,  $CaS_3$ , in the compound, and his analyses lead to the formula



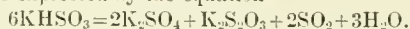
The second oxysulphide is prepared by passing a strong stream of sulphuretted hydrogen through milk of lime for several days, filtering, and allowing the liquid to stand in a loosely-covered vessel for about six months. It is then evaporated down to half its bulk, when much  $SH_2$  is evolved and calcium hydrate deposited; after filtering, the liquid is again allowed to stand in a cool place; the oxysulphide separates out in orange-coloured, prismatic crystals. The author is of opinion that these also contain calcium trisulphide, and assigns to them the formula,  $CaS_3 + 3CaO + 14 \text{ or } 15 H_2O$ .

II. Composition of the crystallised hydrate of sulphurous acid. This compound separates out in colourless crystals when water at  $3^\circ$  to  $4^\circ$  is saturated with sulphur dioxide; there is great difficulty in obtaining it free from adhering ice, so that its composition is not even approximately known. Thus, according to Schönfeld, its formula is  $SO_2 + 15H_2O$ ; according to Pierre,  $SO_2 + 9H_2O$ . The melting-points given by various chemists vary also from  $-2^\circ$  to  $+5^\circ$ . To obtain the pure hydrate, the crystals must be quickly filtered, and submitted to repeated pressing in a room the temperature of which is about  $8^\circ$ . Crystals thus prepared melt at  $+14^\circ$  and have at that temperature the sp. gr. 1.147. Analysis showed that they have the composition,  $SO_2 + 7H_2O$  or  $H_2SO_3 + 6H_2O$ .

III. Decomposition of the Sulphites of Potassium by Heat. The acid sulphite  $KHSO_3$  is completely decom-



posed on heating to  $190^{\circ}$ , water and much sulphurous acid being evolved. The residue has a neutral reaction, and consists of sulphate and hyposulphite. The reaction may be expressed by the equation—



A little free sulphur is also produced, and on stronger heating this sublimes, a residue of brown polysulphide being left. Potassium pyrosulphite  $\text{K}_2\text{S}_2\text{O}_3$  also yields, on heating, a mixture of sulphate and hyposulphite. The neutral sulphite  $\text{K}_2\text{SO}_3 + 2\text{H}_2\text{O}$  is only decomposed at a red heat, producing potassium sulphate and brown polysulphide.—D. E. J.

*Obtaining Sulphur from Sulphuretted Hydrogen.* Eng. Pat. 5070, October 25th, 1883. C. F. Claus, London.

IN Patent 3608, July 29, 1882, the patentee describes a method for producing sulphur from sulphuretted hydrogen, by passing the latter with a quantity of atmospheric oxygen equivalent to its hydrogen through anhydrous oxide of iron. When pure oxide of iron is used the temperature often rises above the point suitable for the operation, and clinkers or fused masses are produced. By the present invention other substances which will not suspend the decomposition of sulphuretted hydrogen are to be mixed with the oxide of iron. The alkaline earths, oxides of chromium, zinc, manganese, copper, or their carbonates or silicates may be used. Whichever of these substances is employed must be intimately mixed with the oxide of iron, made into a stiff paste with water, dried, and broken into pieces of a convenient size. Instead of the oxide of iron, other pyrophoric bodies capable of decomposing sulphuretted hydrogen at an elevated temperature, may be used. Such are the oxide of chromium, chromates, the oxides of cobalt, silver, copper, manganates and permanganates, carbonate or oxide of manganese, or calcined sulphate of copper. If a soluble salt is employed it is advisable to soak in its solution pieces of burnt porous clay, pumice stone, or similar material, to dry these and break them into sizes varying from a walnut to a pea. The oxide of iron or substitute is placed in a layer from six to twelve inches deep on the false bottom of a closed iron tank lined with lead. For gaseous mixtures very poor in sulphuretted hydrogen a much deeper layer is required. Beneath the false bottom are two apertures, by one of which the sulphuretted hydrogen is allowed to enter, and by the other atmospheric air. Above the layer of oxide is an opening in the tank for the escape of the free sulphur formed in the operation.—A. R. D.

*Manufacture of Sulphuretted Hydrogen, etc.* David Urquhart, Westminster. Eng. Pat., No. 5428, of November 17, 1883.

THE materials employed are the sulphates of the alkaline earths, especially sulphates of baryta and strontia. The powdered sulphate is intimately mixed with two equivalents of powdered carbon, and roasted in a reverberatory or muffle furnace. The resultant crude sulphide is spread out, cooled, moistened and broken into lumps, which are placed in a cupola furnace. As soon as the contents of the cupola attain a temperature of from  $370^{\circ}$  to  $470^{\circ}$  centigrade, steam, superheated to about the same temperature, is blown into the furnace through tuyères suitably fixed in the furnace, and rising through the mass decomposes the sulphide, forming a hydrate of barium, etc., and liberating the  $\text{SH}_2$ , which passes through coolers to condense the surplus steam, and thence to burners, where it is converted into  $\text{SO}_2$  and  $\text{H}_2\text{SO}_4$  in the usual manner. The residual  $\text{BaH}_2\text{O}_2$  is tapped and run out of the furnace from time to time.—H. J.

*Improvements in Decomposing Sulphate of Iron and in the Obtaining of Sulphurous Acid and Red Oxide of Iron thereby.* Eng. Pat. 5930, April 4, 1884. Thos. Terrell, London.

GREEN Copperas is mixed with one-tenth of its weight of free sulphur and dried until the water of crystalli-

sation is removed. The resulting pulverulent dry mass is then heated in a kiln, to which a suitable quantity of air is admitted, and which has an outlet into a vitriol chamber. The kiln is heated by a furnace placed underneath. The sulphur reduces the sulphuric acid of the copperas to sulphurous acid, and in this state the decomposition is so rapid that in from an hour to an hour and a half a brilliant red oxide of iron is left alone in the kiln. By this invention a much lower heat and of shorter duration is required than by the process generally in use, while the product obtained is much more free from sulphuric acid.—A. R. D.

*Manufacture of Solid Non-deliquescent Phosphate of Lime, Soluble in Water.* C. D. Abel (a communication from François Barbe, Paris). Eng. Pat., No. 5644 of May 3rd, 1884.

THE inventor claims the production of a new solid phosphate of lime, intermediate between the mono and bi-basic salts, and containing 48 to 50 parts of  $\text{P}_2\text{O}_5$  to from 20 to 23 parts of  $\text{CaO} \cdot \text{H}_2\text{O}$ . These proportions give a solid substance non-deliquescent, soluble in water, very rich in phosphoric acid, of uniform chemical composition, and not capable of acting on the bags in which it is packed. The pure "triple phosphate" of lime should be perfectly soluble in water; any insoluble parts it may contain are due to the presence of iron in the  $\text{P}_2\text{O}_5$  solution employed.—H. J.

## VIII.—GLASS, POTTERY, Etc.

*Manufacture of Porous Pots and Porous Plates for Galvanic Batteries.* T. Coad, London. Eng. Pat. 5195, November 1, 1884.

THE ordinary clay from which porous pots and plates used in batteries are made is mixed with a carbonaceous solution; one consisting of about 10 quarts of vinegar to 14 pounds of sugar is found to answer well, a sufficient quantity being added to render the clay plastic. After being moulded and dried, the pots or plates are packed in a carbonaceous material, *c.g.* plumbago, for the purpose of excluding the atmosphere, and calcined. A second and similar treatment is sometimes pursued. The patentee claims the manufacture as first described, the calcining and baking in a covering of plumbago or carbonaceous matter, and finally the second treatment of such articles in a similar manner. Such pots and plates are stated to have a higher conductivity and are more homogenous, preventing endomose and increasing the constancy of the battery.—C. C. H.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Application of Chemical Agents to Natural or Artificial Stones, and to Stuccoes, in order to diminish their Porosity and increase their resistance to External Influences.* William Spence, London. A Communication from Messrs. Faure and Kessler, of Clermont-Ferrant, France. Eng. Pat. 4954, Oct. 17, 1883.

THIS process consists in impregnating the stucco or plaster with fluorides or fluosilicates of various metals in solution, or otherwise—reactions taking place which leave only insoluble residues in the stone. For impregnating white casts, the fluorides and fluosilicates of ammonium, magnesium, aluminium, zinc and lead are used, and the same salts of iron, manganese, chromium, etc., for coloured articles. The earthy or metallic phosphates dissolved in phosphoric, hydrofluosilicic, or even sulphuric acid are also available for this purpose, together with the alkaline aluminates and silicates.—A. R. D.

## X.—METALLURGY, MINING, Etc.

*Improvements in the Treatment of Copper Mattes in order to obtain the Silver and Gold therefrom.* Frederic Claudet, Coleman Street, London. Eng. Pat. 4818, October 10, 1883.

THE argentiferous matte is to be crushed and calcined till "sweet;" it is then treated with hydrochloric acid, by preference of 1·140 sp. gr. and with constant agitation at about 190°F, until the acid is saturated. The silver and gold in solution are exactly precipitated by a soluble iodide, and may then be recovered by means of iron, zinc, or an alkaline sulphide.—W. G. M.

*The Production of Mineral Phosphates from the Slags resulting from Thomas's Dephosphorising Process, etc.* C. D. Abel, London. A Communication to Johannes Braudt, of Berlin. Eng. Pat. 5000, Oct. 20, 1883. This invention received Provisional Protection only.

THE slag from Thomas's dephosphorising process, or minerals of a similar composition, are slightly broken up and treated with strong liquid or gaseous hydrochloric acid till the iron, manganese and phosphates have been rendered soluble, and the silicates and sulphides susceptible of decomposition have been decomposed. The extract thus obtained is evaporated to dryness, and the residue heated to redness in a current of air, or preferably of steam. By these means the hydrochloric acid is driven off to be recovered, the iron and manganese rendered almost insoluble, and the silica entirely so. The resulting mass may be lixiviated with dilute hydrochloric or sulphuric acid, and the phosphates obtained from this solution, either by evaporation or by precipitation with lime, magnesia, or dolomite. When hydrochloric acid is used for lixiviating, the more readily soluble peroxide of manganese is to be reduced by sulphurous acid, or any other convenient reducing agent. The same end is attained by always keeping an excess of phosphate of lime in lixiviating, as the peroxides are only attacked when all the phosphates are dissolved. The mass left after the lixiviation, being very rich in iron and manganese steel.—A. R. D.

*An Improved Method of Purifying Metallic Copper in the presence of Arsenic or Antimony or both.* Fredk. William Pittuck, Hebburn, Durham, and James Marwood Hucklebridge, Jarrow, Durham. Eng. Pat. 4244, March 3, 1884.

THE purification is effected by blowing dry steam through the melted copper, whereby the arsenic and antimony become converted into volatile compounds, which pass away into the flues; and the steam acts also as a mechanical stirrer, bringing fresh portions of the metal into the sphere of action.—W. G. M.

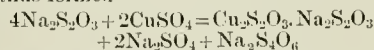
*Improved Process for "Untinning" Tin-plate Refuse in Hermetically closed Apparatus, and at the same time obtaining Ammonia.* Ludolph Poengsen. Eng. Pat. 4684, March 11th, 1884.

THE inventor treats tinned iron scraps in a specially constructed apparatus with a solution containing an alkali and an alkaline nitrate. The tin is thus removed as alkaline stannate, the ammonia which is evolved being passed into water or weak acid. The residue of iron is further treated with dilute hydrochloric acid or solution of ferrie chloride to complete the removal of the tin. A special apparatus for washing the treated iron is also described.—E. J. B.

*Improved Process of and Compound for Removing Precious Metals from Ores and Metallurgical Products.* Edward Hubbard Russell, Park City, Utah, U.S.A. Eng. Pat. 5431. March 25, 1884.

THE object of the inventor is to recover the silver and gold existing in ores or products as metallic silver, as

sulphides of silver or gold, or as compounds of silver with antimony or arsenic, and which cannot be extracted by leaching with sodium hyposulphite alone. The ore, if wished, may be first lixiviated with water and then with hyposulphite in the usual way, and subsequently treated with the new solution; or the latter may be employed alone. This leaching solution consists of an ordinary hyposulphite solution, to which has been added a soluble compound of copper. The sulphate is preferred, but (in descending order of effectiveness) the hyposulphite, chloride, nitrate, carbonate, acetate, cuprous carbonate, ammoniated copper and hydrate may be employed. A double salt of cuprous-sodium-hyposulphite is thus formed—



in which the copper can be replaced by silver, the eliminated copper taking the place of the silver in the compound broken up by the solution. The solution is made by adding 1 to 3 per cent. of the copper salt to a 1 to 5 per cent. solution of the hyposulphite of an alkali or alkali earth. The gold and silver are recovered by precipitation as sulphides.—W. G. M.

*Improvements in separating Ammonia from Blast Furnace and other Gases or Vapours, and in Apparatus therefor.* Gavin Chapman, Glasgow. Eng. Pat. 6406, April 16, 1884.

CONNECTED with the flue leading from the blast or other furnace are chambers or vessels provided with no outlets. In the lower part of each of these is placed a solution of sulphuric or other suitable acid. In each also is a gun metal shaft carrying a number of circular copper plates or discs, small distances apart, and preferably bolted together at points near their circumference. When the shaft is made to revolve, the plates (which dip into the acid solution) continually expose a fresh acid surface to the ammonia, which finds its way in from the flue, and is absorbed.—A. R. D.

## XII.—PAINTS, VARNISHES, AND RESINS.

*Rendering Fubries, Fibrous Materials of all kinds, and Leather Impermeable.* Frank Worth. Eng. Pat. 4921, October 16, 1883.

THE object of this invention, which has received provisional protection only, is to render different materials waterproof by saturating them with a solution of palmitate or oleate of alumina in petroleum, ether, or benzene, which, after the evaporation of the solvent, leaves an impermeable varnish.—C. F. C.

*Manufacture of Anhydrous Sulphide of Zinc.* Edward Hill. Eng. Pat. 4980, October 19, 1883.

THE object of this invention is to prevent the oxidation, hardening, discolouration and loss of body, or covering power, which have attended the manufacture of anhydrous sulphide of zinc as hitherto conducted. Hydrated zinc sulphide, prepared in any convenient way, is, after being washed and partially dried, saturated with a solution of zinc chloride, dried, and ignited. Or a solution of zinc chloride may be partially precipitated with a solution of barium sulphide, the precipitate pressed without washing, in order that it may retain a certain quantity of undecomposed zinc chloride, dried and ignited. The barium chloride solution resulting from the above reaction is decomposed with sulphuric acid, the resulting barium sulphate being utilised as "permanent white," and the solution of hydrochloric acid being used to prepare a fresh quantity of zinc chloride solution. The washed and dried zinc sulphide obtained by either of the above processes is then heated to redness with continual stirring in a reverberatory or muffle furnace, until fumes of zinc chloride begin to be evolved. The charge is then drawn into water. The volatilisation of the zinc chloride prevents access of air, and therefore prevents oxidation and discolouration.—E. J. B.



*Improvements in the Manufacture of White Lead.* H. B. Condy, Battersea, Surrey. Eng. Pat. 1693, Jan. 18, 1884.

THE improvements consist in modifications of the process patented by the inventor, under date March 14, 1881, No. 1095. The inventor finds that white lead, prepared according to his previous process, is liable, under certain conditions, to slight variations of tint, which detract from its value, and he therefore prefers the following process, the subject of the present patent:—Metallic lead is melted, skimmed, and granulated by dropping it into water. The granulated lead is then placed in tanks of suitable construction, when it is alternately exposed to atmospheric action, and to dilute acetic acid (1 grm. acid of 1·045 sp. gr. to 12½ grms. water) at intervals of two to four hours, until the acetate solution attains the specific gravity of 1·040 at 60° F. By this method manufactured metallic impurities present in the lead, such as silver or copper, are left undissolved, and on this account old lead, such as lead piping, roofing, etc., may be profitably utilised for this purpose. The solution of basic acetate of lead thus obtained is treated with bicarbonate of soda in the proportion of 30lbs. for every 60lbs. of acetic acid originally employed, and the solution tested, and more bicarbonate added, if necessary, so as to effect the complete precipitation of the lead. The precipitated white lead is washed, pressed, and dried in the usual way.—H. J.

### XIII.—TANNING, LEATHER, GLUES, SIZES, AND RESINS.

*Manufacture of Glue and Bye-products from Bones, with a Method and Apparatus for utilising such products in the Manufacture of Paper-Pulp.* W. R. Lake, London. Communicated by Carl Valdemar Flodqvist, Mölnådal, Sweden. Eng. Pat. 4356, Sept. 11, 1883.

OF a number of chambers, lined with a mixture of asphaltum and resin, and connected with each other by channels to allow of the passage through the series of sulphurous acid gas, generated in a furnace in which sulphur and pyrites are simultaneously burnt, two are filled with bones freed from fat in the usual manner; the remaining chambers are filled with limestone. The sulphurous acid gas is cooled in a refrigerator before being conducted through the series of chambers, and the latter are protected from the action of the acid gas by the coating of asphaltum and resin. A wooden grating forms the bottom of each chamber, while at the top is in each case a rose connected with a pipe conveying a stream of water from a reservoir. In this way the water is equally divided over the bones and limestone in the chambers, and as it descends it is met by the ascending sulphurous acid gas; when it reaches the bottoms of the chambers it contains in solution acid phosphate of lime or acid sulphite of lime. This fluid is collected and again passed through the chambers, the operation being repeated until the required strength of 5° Beaumé is attained; when this is the case fresh water is allowed to run into the chambers, until a similar solution is obtained. When all the phosphate of lime in the bones has been dissolved, the cartilage remaining behind is taken out and prepared for glue in the usual manner. Any chamber may be emptied and refilled without interrupting the working of the apparatus. According to that part of the invention which relates to the manufacture of paper pulp, a spherical boiler, of steel plates, lined on the interior with lead plates, and resting upon trunnions, is a little more than half filled with sawdust, previously cleansed from bark and dirt, and sufficient of the solution of acid phosphate of lime and acid sulphite of lime, at about 5° Beaumé, to entirely cover the sawdust. The boiler is now turned for half an hour, in order that the solution may be thoroughly absorbed by the sawdust, and at the end of this operation dry steam is admitted through a pipe, the steam pressure being gradually increased to 70lb. per square inch, which

pressure is maintained for from eight to nine hours, when the sawdust will have been converted into paper pulp. Wood, straw, and other fibrous matters may, with certain precautions, be made use of for the manufacture of pulp by this process.—E. G. C.

*Manufacture of Coloured Grained Leather.* Frank Worth. Eng. Pat. 4848, October 11, 1883.

THIS is a communication from Messrs. Hansmann Bros., of Homburg, and obtained provisional protection only. The process is said to be suitable for all kinds of leather, and may be used with all kinds of graining preparations.—A. J. K.

*An Improved Manufacture of Size from Wet Calf, White Leather shavings, or Cuttings.* Isaac Cripps, jun., Brixton. Eng. Pat. 2843, Feb. 6, 1884.

THE shavings are first soaked in water for some hours, and then washed out and allowed to drain. They are now allowed to remain for twelve hours or more in "rather more than their own weight of warm water of a temperature of 120° Fahrenheit or thereabouts, into which there has been previously placed a solution made by dissolving ordinary soda in boiling water in the proportion of about 3½lb. of soda for each hundredweight of shavings or cuttings." The mixture is next well stirred and drawn out upon a screen placed in a slanting position between the top of the vat containing the cuttings or shavings, and the top of a lower vat or tub containing clean cold water. The shavings are pushed down the screen into the lower vat, in which they are well washed previously to being allowed to drain for ten or twelve hours. The cuttings so prepared are now covered with boiling water and boiled for about five hours, this part of the process being carried out in "an ordinary copper or pan provided with a steam coil or jacket bottom, and with an outlet in the lower part to which a tap is fixed." Around the outlet to the tap is placed a wire gauze, cage or trap, filled with clean cow or other hair, through which, after the boiling already described, the size is run off for treatment with alum in the usual manner.—E. G. C.

*Improvements in the Manufacture of Artificial Leather.* Walter Travis. Eng. Pat. 5191, March 20, 1884.

THE object of this invention is the preparation of a felt backing, upon which a japanned surface is formed. For this purpose the felt backing is covered on one side with a coating of oil of japan, mixed to the consistency of a thick paint; this is allowed to dry, and, if necessary, the treatment is repeated more than once. The japan used is from the tree known as the "stigmara verniciifera." The surface, when thoroughly dry, and rendered smooth by rolling or otherwise, is varnished with any suitable elastic varnish.—A. J. K.

### XIV.—AGRICULTURE, MANURES, Etc.

*The Action of Solutions of Sodium Chloride and Zinc Sulphate upon the Growth of Plants and Composition of Soils.* F. Storp, J. König and others. Bied. Centr. 1884, 76.

CHLORIDE of sodium in comparatively large amounts has a specific injurious action upon the growth of many plants, and especially upon the germination of seeds; but in the main its evil effects are due to its comparatively powerful solvent action upon the constituents of the soil, whereby the latter may become not only rapidly impoverished of its plant foods, but also brought into a condition unfavourable for healthy growth, by reason of the considerable amounts of magnesium or calcium chloride formed by the action of sodium chloride upon magnesium carbonate, or calcium sulphate, carbonate or silicate. The waste waters from salines and mineral springs must, therefore, be considered upon the whole to have a prejudicial effect upon agriculture. The question of the influence of compounds of zinc is a very important

one, but has, up to the present, been little investigated. Freytag (Bied. Centr. 1882, 505) considered that solutions of zinc sulphate had a specific poisonous effect upon plant life, but maintained, at the same time, that by reaction with other constituents of the soil, this salt became innocuous. Experiments, however, conducted by König upon the same plan as the above-mentioned researches with chloride of sodium, have led to the following deductions: Even dilute solutions of zinc sulphate applied to the roots of assimilating plants, induce sickness and death with a rapidity proportionate to the concentration of the solution. To many fungi and germinating embryos which have not commenced to assimilate, zinc sulphate appears to be indifferent. The specific poisonous action of zinc sulphate appears to be destroyed by mutual reaction between it and the soil constituents; but such reaction is attended by changes in the soil by no means beneficial to plant life. In consequence of the mutual decompositions which take place between zinc sulphate upon the one hand, and the carbonates, phosphates, silicates and humates of potassium, calcium and magnesium upon the other, the basic plant foods are rendered so easily soluble that the soil rapidly becomes exhausted. Moreover, the bases originally combined with carbonic, silicic, phosphoric, etc., acids now occur as sulphates, a form of combination indicated by experience as one of the least efficient. It is also probable that the plant foods of an acid nature, such as humic acid, form insoluble zinc compounds, and thus are not readily assimilable. Further experiments made in 1882 by König, Cosack, Böhmer and Weigmann, upon a larger scale, confirmed the above. The authors consider that a water containing 1 gramm of sodium chloride per litre may be expected to exert a prejudicial effect upon soils, whilst with zinc sulphate a very much smaller degree of concentration acts similarly. Experiments with ferrous sulphate shewed that besides an exhausting influence similar to that of sodium chloride and zinc sulphate, it possesses the property of closing up the pores of the earth after a short time, and so gives rise to a "sour" condition of the soil.—W. D. B.

*Carnallite as a cheaper substitute for Kainite for manurial purposes.* Troschke. Bied. Centr. 1884, 98.

THE well-known valuable property which kainite possesses of absorbing considerable amounts of volatile ammonia compounds and salts, and which has caused it to be employed for admixture with stable manure, is shared by carnallite, and that, according to the author, in an even greater degree. He found, indeed, that under the same conditions, kainite absorbed 4.5 per cent. of its weight of ammonia from an ammoniacal atmosphere, whilst with carnallite the amount was 9 per cent. He calculates that for ammonia fixation, carnallite will be more than three times as cheap as kainite. It remains to be seen whether the larger proportion of chlorides and magnesium compounds in carnallite than in kainite will have any injurious influence upon plant life.—W. D. B.

*Manuring a Crop of Rye with Kainite and Bone Meal.* F. W. Steffens. Bied. Centr. 1884, 99.

A FIELD of well-marled arable land with sandy soil, 20 acres in area, was, after ploughing in the clover, dressed with 300lb. of kainite, and later, at the time of seed planting, with 100lb. of bone-meal. An area of one-tenth of an acre was left unmanured, and the crop from this compared with the crop from one-tenth of an acre of the manured portion. The yields were as under:—

$\frac{1}{10}$ acre manured.	$\frac{1}{10}$ acre unmanured.
104 kg haulms	80.8 kg haulms
61.5 " corn	44.2 " corn
2.5 " chaff	2.2 " chaff
171.0 total.	127.8 total.

The cost of manuring was 14.4 \*marks per acre, the value of the increase of the manured crop 39.38 marks, so that the manuring effected a clear gain of 24.98 marks. The application of 100lb. of bone-meal in a previous year, unaccompanied by kainite, was by no means so efficient.—W. D. B.

*Manurial Experiments with Sea-Mud and with a Moor-Compost.* Enckhausen. Bied. Centr. 1884, 100.

A piece of poor, sandy arable land, with cold impervious sub-soil, was divided into three plots of 15sq.m. each in area. The first plot was manured with 60kg. of sea mud collected from an arm of the sea, the tide having just receded, the second plot with 60kg. of decomposed stable dung, and the third was left unmanured. The following table gives the results of the experiment, the yields being expressed in terms of kilogrammes per hectare.

No.	Manure.	Spring Rye.		Oats.		Potatoes.
		Corn.	Husk.	Corn.	Husk.	
1	Sea-Mud....	2500	4000	2666	2800	32000
2	Stable Dung	1883	3000	2333	2333	28666
3	Unmanured	1500	3000	1666	2000	23333

These figures shew that the sea-mud in all cases caused a higher production than when the ground remained unmanured or manured with stable dung. The mud when collected had not settled down and so contained a high percentage of water, in fact a load of 200 \*centners only contained from 77 to 78 centners of dry substance.

In spite of this, the author believes that its use will be attended by economy.

Experiments were also made as to the fertilisation of a piece of moor-land reclaimed from forest 30 years since. Before the application of the compost about to be described, the annual yield of potatoes was only about 3600kg. per hectare. A compost was prepared of

6 loads sea-mud, equivalent to 470 centners dry substances

457 Centners of stable dung

40 Centners phosphorite meal

40 Centners of potassium and magnesium sulphate

7 Loads of marl, and some sawdust

The yield per hectare after the application of the above was 11,893kg. of potatoes.

With summer green crops such as the cereals the above compost gave much better results than stable dung and its cost was less. With crops such as potatoes there was little difference between the results of the two dressings. The cost of the compost per centner was estimated at 0.24 \*marks, that of the stable dung at 0.40 marks.—W. D. B.

*Manuring Experiments with Common Salt and Herring Waste.* E. H. d'Orval and A. Pagnoul. Bied. Centr. 1884, 92.

THE authors find that in quantities not exceeding 40kg. per hectare, sodium chloride exerts neither a favourable nor unfavourable influence upon the growth of cereal crops, and that in small amounts it may therefore be added without danger to guano to fix volatile constituents therein. The authors have also made experiments as to the value of salted herring waste for manuring purposes. They found that the composition of this was by no means constant, as the following figures shew:—(1) Composition of a sample of herring waste analysed five to six years ago. (2) Of a sample analysed recently. (3) According to Nantier.

	(1)	(2)	(3)
Water .....	14.8	39.53	20.55
Nitrogen .....	4.3	1.95	1.26
Phosphoric Acid..	4.7	1.95	1.19
Sodium Chloride..	22.5	40.36	52.00
Other Organic Substances .....	35.2	11.77	25.00
Other Inorganic Substances....	18.5	4.39	
	100.0	100.00	100.00

Further investigations were made by the authors in the autumn of last year upon a fresh sample of herring



waste from Boulogne. The following are the analytical results :

	Fresh Waste.	Dried Waste.	Waste washed and dried.
Water .....	47.7	1.6	2.1
Nitrogen .....	2.4	4.6	5.3
Fatty matters .....	12.1	22.7	36.8
Other Organic Substances .....	13.1	24.7	20.0
Sodium Chloride .....	18.0	33.9	10.3
Phosphoric Acid .....	2.3	4.3	6.7
Other Mineral matters .....	4.3	8.2	18.8

Practical trials with various crops shewed that, as was to be anticipated, the original waste had a most injurious influence upon the yield, owing to the enormous quantity of sodium chloride which was unavoidably applied to the soil if the whole supply of nitrogen and phosphoric acid was to be derived from the waste. However, on allowing a period of time to elapse between the application of the waste and the sowing of the seed, sufficient for the greater portion of the sodium chloride to be washed out of the soil, much better results were obtained. The analysis above quoted, of the waste washed and dried, shews that the sodium chloride can be removed by water to a considerable extent without any practical loss of nitrogen and phosphoric acid. The manurial value of the washed and dried waste the author places at 12 marks per centner.—W. D. B.

*African Guano.* P. Wagner. Bied. Centr. 1883, 849.

THE author has obtained the following analytical results with a sample of African Guano :—

Nitrogen ..	In the form of nitric acid ..	0.02%	14.68
	ammonia ..	4.57	
	In organic compounds, uric acid, Guanine, &c. ....	10.09%	
Phosphoric Acid ....	Easily soluble in water .....	3.05%	9.75
	Insoluble in water .....	6.70%	
Potash .....			4.65

*The Influence of temperature upon the growth of Plants.* Hellriegel. Bied. Centr. 1884, 123.

FOR every plant and for every physiological function thereof, there are certain limits of temperature between which the processes take place in a normal manner. As the lower limit is approached, the processes of life become gradually more feeble and finally cease, whilst on approaching the higher limit the processes become gradually more vigorous until that limit is reached. At temperatures above this point the processes again become feeble, and a third degree of temperature is at last reached at which life is impossible. The temperature most favourable for the normal exercise of the various physiological functions lies between 20 and 40° C., between these degrees there is an optimum for each plant. Temperatures above 50° C. are unfavourable to plant life. The author, in this article, gives the results of his experiments upon the influence of low temperatures on germination of seeds of the winter rye, winter wheat, barley, oats, maize, rape, linseed, vetch, pea, horse-bean, lupine, clover, buckwheat, asparagus, red-beet, cress, and cucumber. The seeds of the majority of our cultivated plants were found to germinate at temperatures below 4° C. of the cereals, winter rye appeared to require the least amount of heat for germination. At 0° C., it germinated in a perfectly normal manner, and was able at this temperature properly to develop its seed leaves and rootlets. Winter wheat was also able to germinate at 0° C., but more slowly than was the case with rye. The summer cereals, barley and oats, appeared to require a higher temperature. At 0° C., a development of the rootlets but not of the seed leaves took place, in fact, even after six weeks the latter had not yet burst through the integuments. At 2° C., and in six weeks, the seed leaves of barley attained, upon an average, to a length of 20mm., of oats to a length of 17mm. Maize was found to require, at least, a temperature of 8.7° C. for incipient germination, at which point, higher than that required by any other cereal, the germinating processes were

extremely feeble. Of seeds with oily albumen, the rape was found to be able to germinate freely at about 0° C., for growth and assimilation, however, a higher temperature was requisite. Linseed shewed at 0° C. no signs of germination, at 2° C. there was a development of the rootlets, but not of the seed leaves. Of the *Papilionaceae*, the vetch required the lowest temperature. At 0° C., both seed leaves and rootlets, were developed, and attained in six weeks to a length of 10mm. For the same degree of development, peas and clover required a temperature of 12° C., and with beans and lupines, it was found that seed leaves, as well as rootlets, would not burst through the integuments below 3° C. At 2° C. asparagus sent forth both rootlets and seed leaves. The lowest temperature for germination of carrot seed was found to be 3° C., for red-beet 5° C. Among the plants experimented upon, the cucumber was found to require the highest temperature. At 8.7° C., the seed gave no sign of life whatsoever.—W. D. B.

(To be Continued.)

*Observations as to the influence of Climate upon the Duration of, and Processes of, the Life of Plants.* F. Hildebrand. Bied. Centr. 1884, 138.

THE author here records a series of observations, made during the year 1882, of the numerous abnormal appearances in the growth of, and discharge of the functions of, various plants, induced by the abnormal climatic conditions obtaining in that period. Seeds of annual plants, which usually remain at rest during the winter before germinating, germinated in the same summer as they were produced, and brought forth physiologically complete plants. Other annual growths did not die down after blooming, but evolved a second inflorescence, the stems at the same time becoming woody. In short it appeared a decided tendency of many short-lived once-flowering plants, to become, under favourable climatic influences, longer lived, and to flower more than once. Certain biennial plants, such as *Digitalis purpurea* evolved, after the first inflorescence, shoots upon the leaf-bearing axis, and shewed a tendency to become shrubs. With other biennials two periods of inflorescence and fruit-bearing were frequently observed together with lignification of the leaf-bearing axis. Many perennials, usually blooming in the spring, flowered in the preceding autumn, and shrubs which generally flower in the spring time, being then bare of leaves, were observed to flower in the preceding autumn, the plant being then covered with leaves. Numerous examples also occurred when the case was exactly reversed. Then again, there were observed cases where shrubs usually flowering once only in the year, had two periods of inflorescence in 1882. Some explanation is thus afforded of the fact that under certain climatic conditions many shrubs, such as *Rhamnus Frangula*, normally flower twice in the year.—W. D. B.

*Manurial Experiments with Potatoes.* M. Mäcker. Bied. Centr. 1884, 102.

THREE series of experiments upon as many varieties of soil were undertaken, special reference being had to the most efficient form in which nitrogen could be applied, to the behaviour, harmful or otherwise, of large amounts of potassium compounds, the comparative efficiency of soluble phosphoric acid derived from faeces or artificial superphosphates, and to the most advantageous proportion between nitrogen and phosphoric acid. The results from the three soils agreed most closely with each other. It was found that in a nearly sterile sand, not more than 8kg. of nitrogen and 10kg. of phosphoric acid were required, that nitrogen in presence of phosphoric acid was more efficient when applied as an ammonium salt than as sodium nitrate, and that ammonium salts alone, increased the yield to the same extent that sodium nitrate alone, decreased it. Potassium salts applied in the spring gave most unfavourable results. Phosphoric acid derived from faeces appeared more efficient than when applied as superphosphate. The second series was made upon a poorly cultivated soil, with the following results: Potassium salts, together with phosphoric acid applied in the autumn caused a decreased total yield but a higher percentage of

starch, but applied in the spring, both quality and quantity suffered. The most favourable ratio between nitrogen and phosphoric acid was found to be 7 : 9.5. With a manuring of potassium salts alone the yield was worse than from the unmanured soil whether these salts were applied in the autumn or spring. The third series of experiments was made upon a light sandy soil brought into cultivation by means of turf and stable dung. With an application of potassium sulphate in the autumn, the total yield was decreased, but the quality improved; when this, however was applied in the spring, the conditions were exactly the reverse. The highest total yield and best results as to percentage of starch were obtained when the ratio between nitrogen and phosphoric acid was 1:1. As before potassium salts alone gave unfavourable results. Applied in the autumn, potassium salts appear to possess a property—not to be under-estimated—of rendering the potato plant less liable to disease and also of improving the shape and durability of the tubers.—W. D. B.

*Practical Trials of the Value of different Varieties of Sugar Beet.* M. Märcker. Bied. Cent. 1884, 126.

As in previous years the author has made a series of comparative experiments as to the value of different varieties of sugar beet grown in Saxony, and as far as possible under the same conditions of manure, etc. The following table sets forth the most important results. The plants were grown in rows of 11 inches wide :—

Variety of Beet.	Yield expressed in centners per morgen.	Sugar in the beet %	Sugar in the juice, %	Co-efficient of purity.	Yield of sugar expressed in centners per morgen.
Kl. Wanzlebener Original	228	11.7	12.7	81.0	26.68
Dippe's Wanzlebener Nachzucht	218	12.0	13.6	85.0	26.16
Kl. Wanzlebener Mohringen	216	11.4	12.7	83.1	24.62
Bestehorn's Imperator	210	11.2	12.3	82.4	23.52
Vilmorin blanche améliorée (Original)	183	12.9	14.1	85.8	23.61
Simon Legrand de mères blanches ....	211	11.7	12.7	83.4	28.20

In the original paper, results of experiments with many other varieties are recorded. The author remarks that no variety experimented upon has proved to be essentially bad.—W. D. B.

*Culture of the Potato.* Cimbal, Nitykowski, Sattig and others. Bied. Centr. 1883, 828.

CIMBAL records trials made with 38 varieties in 1882. The soil was loamy and rich in humus, the sub-soil clay. A dressing of superphosphate and stable manure was applied. As regards starch production, Aurora gave the best results, Richter's Imperator the second best, and Snowflake the worst. It was also found that the finest yields were obtained from whole large seed tubers, and that generally speaking whole seed tubers gave a better total yield, and that richer in starch than when divided. Nitykowski has experimented with some 37 varieties in a sandy loam soil dressed with stable manure. Richter's Imperator gave the best, Eos the second best results, and Snowflake the worst. Sattig-Würchwitz finds that different varieties of potato behave very differently in various soils. In a light, slightly manured potato soil, Richter's Schneerose gave the best, Seed Gleason the second best, and Magnum Bonum the worst results, whilst in a heavy, strongly-manured sugar beet soil, Seed Gleason gave the best, Snowflake the second best, Richter's Long-Bearing the worst results. The variety Dabers was the most diseased. Upon the whole the heavy soil appeared most favourable. Kennemann-Klenka obtained the finest yield of tubers with Yellow Rose variety, but the highest percentage of starch with the Champion. J. Timm-Papenhoe records observations of 24 varieties grown in marshy and sandy loam soils. In the first case Late Rose and Euphylllos gave the best yield of tubers, in the second

Lippische's Rose and early from Chicago, the worst respectively being Prima Donna and Queen's Potato. Gieth has cultivated 29 varieties in a damp, sometimes wet, well-manured clayey loam soil with clay sub-soil. The variety Andersen gave the highest yield of tubers and starch, and Trophime the next highest percentage of starch in the tuber. Heine-Emersleben cultivated in 1881, in a well-manured, calcareous sugar-beet soil, 52 varieties. Lippische's Rose gave the highest yield of tubers and starch, and the second highest percentage of starch in the tubers. The variety Alcohol gave the highest percentage of starch in the tubers, and in connection with this the author remarks that the varieties Eos, Aurora, and Alcohol may be depended upon to give good yields of starch, and are thus suitable for spirit manufacturers. This observation is confirmed by five years' experience. Among new varieties, the author finds Richter's Long White to be the most prolific. The following figures as to the proportion between crop and seed are due to Lampert :—

Variety.	Yield in kg. from 124 kg. seed tubers.
Champion .....	437
Anderson .....	377
Aurora .....	295
Lippische Rose .....	295
Imperator .....	252
Flour Ball .....	195
Purple Red .....	192
Early King .....	160
Snowflake .....	90

Among novelties, the variety White Elephant, obtained by crossing White Peachblow with Chili Garnet, is extraordinarily prolific, attains to a length of 27cm., and must be favourably noticed. It is stated to resist frost very well. Experiments have recently been made at Brest on the cultivation of the Ohrend-Potato from the island of Goritti (Uruguay). It appears to be hardy and prolific, and may give good results if crossed with some cultivated variety. Jean Pilorgé, of Bretagne, contributes his results with 42 varieties in connection with the proportion between seed and yield.

With 2500 kilos of seed.

Champion of Scotland yielded .....	41,650 kg
Van der Vere .....	38,050 "
Seguin .....	33,317 "
Magnum Bonum .....	32,200 "
Boule Depin .....	33,183 "

The first, third and fourth varieties are healthy, and may be stored safely, the second and fifth leave much to be desired in these particulars. The following tabular statement records the most important results of Emel's comparative trials of the value of whole seed tubers and of fragments, containing each a bud :—

24 large whole tubers .....	26.0kg. }	66kg.
24 medium " " .....	22.0 " }	
24 small " " .....	18.0 " }	
24 large fragments with buds above .....	23.0kg. }	60kg.
24 medium " " " " " " .....	19.5 " }	
24 small " " " " " " .....	17.5 " }	
24 large fragments with buds below .....	27.0kg. }	67kg.
24 medium " " " " " " .....	21.0 " }	
24 small " " " " " " .....	19.0 " }	

Four concordant series of observations on different varieties of potatoes enable the author, A. Leydecker, to make the following deductions as to the practical efficiency of the variously situated buds of the tuber. (1) The vegetative efficiency of the buds upon a tuber gradually decreases from the summit to the base. (2) The buds upon the summit of the tuber yield the largest crops, and the relatively greatest amount of large tubers, whilst the reverse is the case with the terminal buds. (3) The total yield is largely dependent upon the amount of substance in the mother tuber, hence a bud united to a tuber may give a production more than twice as great as a similar bud separated from the tuber would.—W. D. B.

*Contributions to the more exact knowledge of the Efficiency of Manures.* By Paul Wagner and others. Bied. Centr. 1883, 729—744, and 798—818.

A CAREFUL series of investigations, extending from the year 1877 to 1882, has led to the development of a method



of research capable of as great accuracy as the best analytical methods, such, for example, as the estimation of phosphoric acid by the molybdate process, and by the aid of which the interesting and important facts in relation to plant foods, detailed below, may be considered established in a trustworthy manner. The earliest trials were made upon a piece of land which had lain fallow for eight years. Forty plots, some of 1 square metre, others of 2.25 square metres, were divided off by underground brick walls .55 metre deep. The soil, which was sandy, was carefully averaged by turning it over to a depth of .55 metre from the surface, and the manure uniformly applied by mixing with the upper layers a known quantity to each plot. Precautions were taken to ensure regularity in the number of seeds sown, and around each plot was planted a strip of barley .5 metre wide to equalize the conditions of light and shade. With crops of potatoes and barley the results were by no means satisfactory. In the former case, two plots manured in the same manner gave yields varying from 22.6 below, to 15.5 per cent. of the yield above the average. In the latter case the yields of barley corn varied from 15.5 per cent. below to 11.8 per cent. above the average. Larger yields were obtained from the eastern than from the western plots. It was considered not improbable that the variations in the state of humidity of the soils, permitted by the perviousness of the brick walls to water, were amongst the more important of the disturbing factors. To eliminate these, the surface soil was completely removed, the walls were coated with cement, and the plots filled in with new soil from the surrounding land. As before, barley was planted in strips round each plot. As regards uniformity of results, a marked improvement took place in the potato crop, and the maximum variation per cent. of the total yield of six plots only amounted to 0.9. With barley, however, the difference between the results of two experiments of the same nature was still very considerable, and several modifications were adopted, but in no case did the maximum error sink below 4.4%, whilst the author had fixed upon 1% as the widest permissible variation. Experiments with potatoes and barley were also made on open land but were unsuccessful, and the author considers that from the unavoidable variations in the moisture of open soils such a method is calculated to yield fallacious results. In 1879 trials were made as to the applicability of small glass or zinc vessels, with encouraging success. With such it was found possible to attain to great accuracy in respect to quantity and quality of soil and manure, an accuracy which would more than counterbalance any variations arising from the smallness of the amount of seed in each experiment. Recourse was first had to cylindrical boxes of zinc plate, 35cm. in diameter, 60cm. high, with perforated bottoms. These were filled with a 5cm. layer of flint, a 30cm. layer of sterile quartz sand, and then a 20cm. layer of moist soil mixed with the manure, in which the barley was sown at a depth of 3cm. The zinc boxes were embedded in the earth, resting upon a layer of flint, and in dry weather their contents were moistened with known amounts of water. With this arrangement, the greatest variation from the average yield of barley was 1.1% of the total yield—a marked improvement upon foregoing experiments. For the purpose of establishing the efficiency of a manure by means of comparative experiments it is of the utmost importance that all the factors of growth—with the exception of that supplied by the manure—be equal and present in excess, whilst the special food supplied by the manure is present only in a relatively minimum amount. This being granted, it is indispensable in a series of comparative experiments that the amount of moisture should not only be maintained in excess of its requirement, but should likewise be maintained at as uniform a point as possible. As the most favourable form of apparatus to attain these ends, a modification of the earlier zinc cylinder was adopted in 1882. In the new cylinder 50cm. high and 25cm. in diameter, the sides were perforated for some distance from the bottom, and an annular vessel, 6cm. wide and 9cm. high, fitted round the perforated portion. In-flow and out-flow pipes enabled the annular vessel to be filled daily with water, and

also to carry off any rain water as soon as the level of the latter rose above a height of 45cm. The arrangement of the contents of cylinder was as under:—First came a 4cm. layer of coarse, and a 1cm. layer of fine particles of coke, then a 27cm. layer of carefully averaged and sifted soil, then a 9cm. layer of a mixture of the soil and manure, and, finally, a 9cm. layer of soil, care being taken that, in all comparative experiments, equal weights of soil occupied equal bulk. It should also be mentioned that the inferior layers were much more firmly compressed than the superior. The average contents of moisture in the soil of these experimental vessels was 17.59, and care was taken that this factor should never sink below 17 per cent. In each vessel 24 seeds were sown at a depth of 3cm., and, at an early period, the number of plants was reduced to 20. An easily adjusted wire frame provided the plants with support. The zinc cylinders not embedded in the ground were arranged in two parallel rows running from east to west. The distance between two adjacent cylinders was 19cm., that between the rows 220cm. The cylinders themselves were protected from the southern sun by a horizontal plank, but, with this exception, were perfectly and uniformly exposed to all changes of weather. Excellent results were obtained by this method, and, in two series of experiments quoted, the greatest variations in the yield were respectively 3.2% and 2.8%. This being so, the author considers that, by carrying on each experiment in six adjacent parallel cylinders, the average of the six results may be regarded as practically free from error. Before passing on to the practical results obtained by the aid of the above method a few points in connection with the manure must be noted. Firstly, as to its amount. It is obvious that a sufficient quantity of the plant-food under investigation must be supplied to ensure normal growth, but in the comparison of two different forms of the same plant-food, care must be taken that the relatively minimum amount only be applied, for should there be an excess, the more efficient will only appear to have performed the same work as the less efficient, whereas, had the minimum been applied, the more efficient would have been utilised to its fullest extent, as also the less efficient, and the difference would be shewn in the yield. Secondly, as to the influence of the less important constituents of a manure. The author here points out that certain compounds, such as gypsum, which are usually regarded as indifferent, must, in certain cases, be held to exert negative action upon the efficiency of the more important plant-foods. He instances experiments as to the depressing effect which gypsum exerts upon the soluble phosphoric acid of a manure. Thirdly, as to the state of subdivision in which a manure is applied. This will be considered in detail below, but the author here asserts the proposition that the maximum efficiency of a plant-food is, under certain circumstances, to be obtained by a certain degree of subdivision in which it is applied, to depart from which point will result in a waste of the food. The author insists upon the necessity, for the above methods to obtain tolerably exact results in manuring experiments, for all conditions in comparative experiments being the same throughout, the most important factor being the amount of moisture in the soil, and lastly for the determinations of the errors of experiment. The author prefaces the record of his practical experiments by arguing that, with different plants, the results of manuring trials cannot be interpreted in the same way. Crops of buckwheat, peas, oats, and vetch, were treated in the same way with a phosphatic manure. In the first case the yield was even less than from an unmanured soil, in the others the total yield was increased. However, in the first, the comparative increase in the amounts of phosphoric acid and proteid substances was greater than in the others. In 1882 some very extended experiments as to the comparative efficiency of precipitated calcium phosphate, potassium phosphate, and soluble phosphoric acid, in the form of superphosphate, were undertaken, each experiment being repeated six times. In some of these, nitrogen, in the form of sodium nitrate, and in the proportion of 50kg. per hectare, and potassium in the form of potassium chloride, and in the proportion of 200kg. per

hectare were supplied. One trial was made to ascertain the extent of the depressing action of gypsum, and so to be in a position to apply a certain correction on account of the gypsum derived from the superphosphate. The following table contains the details of these experiments. The crop was of peas:—

trials 10 to 15 when gradually increasing amounts of phosphoric acid were applied, the total phosphoric acid in seed and haulms together remained very constant, but that an increased supply of this food was followed by decreased assimilation of the same by the seeds, an increased amount being absorbed by the haulms. The

No. of Trial	Description of Manure.	Height of Plant. That of the unmanured = 100.		Yield of Seed and pod grms.	Yield of Seed and pod. The unmanured = 100.	Yield of		Proportion of Seed to 100 parts of pod.
		June 16.	June 21.			Seed grms.	Pods grms.	
1	None.....	100	100	182.1	100.0	71.6	107.8	69
2	Nitrogen from $\text{NaNO}_3$ .....	102	101	185.1	101.5	77.6	107.5	72
9		102	101	185.3	101.5	76.3	103.0	70
3	Potassium from $\text{KCl}$ .....	107	101	191.6	105.0	77.3	111.3	68
4	150 kg. per hectare of $\text{P}_2\text{O}_5$ soluble in water.....	105	103	206.5	113.2	81.3	125.2	65
5	Nitrogen and Potassium from $\text{NaNO}_3$ and $\text{KCl}$ respectively.....	107	107	202.3	110.9	81.0	118.3	70
6		125	116	210.9	132.1	95.3	115.6	65
7	Phosphoric Acid soluble in water 150kg. per hectare, Potassium and Nitrogen.....	128	120	250.9	137.5	100.5	150.1	66
8	Potassium Phosphate (150kg. $\text{P}_2\text{O}_5$ per hectare) and Nitrogen.....	125	116	250.1	137.3	99.3	151.1	66
10	150kg. $\text{P}_2\text{O}_5$ Soluble Phosphoric Acid and Nitrogen.....	105	105	211.7	116.0	81.8	126.9	67
11	150kg. Phosphoric Acid soluble in Citrate Solution, and Nitrogen.....	106	105	216.5	118.7	83.1	133.4	62
12	300kg. Soluble Phosphoric Acid, and Nitrogen.....	105	104	201.4	112.6	77.7	126.7	61
13	300kg. Phosphoric Acid soluble in Citrate Solution, and Nitrogen.....	106	101	206.2	113.0	73.9	132.2	57
14	450kg. Soluble Phosphoric Acid, and Nitrogen.....	105	101	195.3	107.1	68.7	126.6	51
15	450kg. Phosphoric Acid soluble in Citrate Solution, and Nitrogen.....	105	101	200.6	109.9	71.9	128.7	56
16	Gypsum.....	98	100	173.0	91.8	70.0	103.3	68

In the cases of phosphatic manuring, the plants had strong stems and healthy leaves, but there appeared to be a tendency to premature decay. A nitrogenous manuring alone effected but slight increase in the yield, but the addition of potassium chloride gave very favourable results. Phosphoric acid from superphosphate, together with potassium from the chloride gave excellent results, and practically the same as an equivalent quantity of potassium phosphate. It will be observed that in trials 10 to 15 the proportion of pods to seed increases with the addition of phosphoric acid. In fact, these results generally point to the conclusion that an increased production of seed is not to be obtained by increasing the amount of phosphoric acid. The author considers that the latter acts as a sudden stimulus to the earlier vegetative processes, but that the period of functional activity of the plant organs is considerably shortened. Determinations of the amounts of phosphoric acid absorbed by the seeds and pods in the above trials were also made, and led to the somewhat remarkable fact that, in

same remarks apply to the formation of protein ( $\text{N} \times 6.25$ ) substances in trials 10 to 15. These observations incline the author to take by no means such favourable views as is customary, of the action of phosphoric acid in assisting the fruit formation and maturing of certain plants, but he considers that the evil effects of phosphatic manures, such as mentioned above, may be counteracted by the co-application of nitrogen and potassium. As it is apparent that the amounts of phosphoric acid applied in these trials were by far in excess of the relative minimum, it was considered necessary to undertake a second series of trials specially avoiding such excess. In this series, undertaken in 1882, the undermentioned varied amounts of phosphoric acid applied were accompanied in all cases by nitrogen 20kg. per hectare, one-half derived from ammonium sulphate, the other from sodium nitrate, and potassium to the extent of 178.6 kilos per hectare derived from potassium chloride. Gypsum was also applied to enable comparisons to be made with superphosphates.

Kilogrammes of Phosphoric Acid per hectare.	(a)	(b)	(c)		Difference between (a) and (b) in parts per cent. of (b).
	Average weight of dry substance in the yield.	Yield of dry sub- stance expressed in parts per cent. of the unmanured crop	Yield of dry substance.		
			(a) Calculated.	(b) Actually ob- tained.	
0	grms.	100.0	—	—	—
40	71.00	114.3	80.6	81.2	0.7
55	81.00	118.3	81.2	81.0	0.2
76	87.98	123.8	87.9	88.0	0.1
85	91.87	129.4	91.5	92.0	0.6
100	94.97	133.8	95.1	95.0	0.1
115	98.01	138.0	98.7	98.0	0.7
130	97.80	137.7	98.7	97.8	0.9



The results obtained were most concordant, and fully justify confidence in the method of experiment. From his trial with crops of peas, and leaving out of consideration any questions as to the form in which the phosphoric acid was applied, the author considers it proved, that so long as an increased application of phosphoric acid increases the yield of the crop there is a constant and exact ratio between the increments of manure and yield. When, however, the point of maximum yield is reached, further additions of phosphoric acid will have not only an indifferent action, but even an unfavourable one. The widest variation between actual and calculated results was only 0.7%. Making allowance for the depressing effect of gypsum, it was found that the different efficiency of the phosphoric acid was independent of the form in which it was applied, and that superphosphates gave better results when coarsely than when finely divided, a condition reversed in the case of the phosphoric acid being in the insoluble condition. The application of phosphoric acid to the crop of peas caused an increase in protein substances and phosphoric acid, and that, independently of the form of combination. Excessive manuring with phosphoric acid will, however, cause a decrease in the protein substances. The same year, and in the same cylinders, without a further addition of manure, experiments were made with crops of barley and vetches, and further confirmation of the correctness of the above observations was obtained. The following will give some idea of the efficiency of the different combinations of phosphoric acid.

#### EFFICIENCY OF PHOSPHORIC ACID.

	Soluble in water, from Super-phosphate	Soluble in Citrate solution from precip. phosphate of lime.	In washed-out Super-phosphate.	Free, and as potassium phosphate.
Correction applied for the action of the gypsum. }	100	.... 100	.... 99	.... 100
No Correction for the gypsum. }	100	.... 104	.... 80	.... 103

As regards the state of sub-division, it was found that coarsely granular precipitated calcium phosphate gave only half the increase of yield as the same compound in a powdery condition did, whilst on the other hand, coarse superphosphate gave an increase greater by one half than the same manure finely divided. Manuring with phosphoric acid alone, appears to cause a very luxuriant vegetation in the youth of the plant, but the co-application of sodium nitrate and potassium chloride favour the more regular absorption of phosphoric acid from the soil during the whole period of growth. To a certain point, application of phosphoric acid increases the total protein substances, but an excessive manuring, whilst causing an increase in the nitrogenous contents of the haulms, has precisely the opposite effect upon the seeds. In sandy soils, and during dry weather, the application of phosphatic manures, especially when accompanied by Chili saltpetre, to potatoes and oats has been attended with very unfavourable results, whilst, under ordinary circumstances, such often causes a premature decay, without having forwarded the appearance of bloom, or formation of seed. As has been shewn above, an excess of phosphoric acid is positively injurious to normal growth.

(b) Trials were made in the year 1877 as to value of potassium salts—with the exception of the nitrate—in improving the quality and quantity of potato crops. They were found to be without any action whatsoever.

(c) In 1879 comparative trials were made of the efficiency of nitrogen derived from saltpetre and from ammonium sulphate in improving the yield of potato crops. Nitrogen, combined as sodium nitrate gave excellent results, but as ammonium sulphate, appeared to be inactive, or even injurious in the earlier periods of growth. Phosphatic applications were likewise without effect.

(d) A series of experiments upon the specific manurial requirements of peas, vetches, barley, and oats was also undertaken by the author, the small zinc cylinders being employed. The crops of vetches and oats so suffered from the weather that no reliance could be placed

upon these results. The following is a summary of the observations upon peas and barley:—

Manure.	Yield of	
	Peas.	Barley.
None .....	100	100
(a) Nitrogen from $\text{NaNO}_3$ 10kg per hectare .....	104	113
(b) Potassium, 80kg per hectare, from KCl .....	100	107
(c) Phosphoric acid, 100kg per hectare, from Superphosphate .....	126	113
(d) Phosphoric acid as in (c) and Nitrogen as in (a) .....	132	116
(e) Nitrogen as in (a) and Potassium as in (b) .....	102	121
(f) Potassium as in (b) and Phosphoric acid as in (c) .....	147	126
(g) Potassium as in (b), nitrogen as in (a), and Phosphoric acid as in (c) .....	151	181

These results speak for themselves, and in the case of the crop of peas confirm the observations made by Schultze-Lupitz upon a larger scale as to the value of a manure supplying potassium and phosphoric acid. In conclusion, the author insists upon the importance of a more extended acquaintance with the specific manurial requirements of our cultivated plants.—W. D. B.

#### The Value of Woody Fibre in Animal Nutrition. M. Märcker. Bied. Centr. 1884, 136.

THE author maintains that woody fibre is for the most part not digestible in the true sense of the word, but that the difference between the amount ingested in the food and that excreted in the faeces has not been assimilated, but has, during the processes of digestion, undergone the so-called marsh-gas fermentation. The author, therefore, considers that only the truly assimilable nitrogen free extractives should be taken into account in calculations of the nutritive value of a given food-stuff.—W. D. B.

#### Cocoa-nut Meal as a Food for Horses. Bied. Centr. 1884, 135.

NUTRITION experiments upon five horses of a Eurasier regiment have been conducted at the instance of the *Société Agricole* of Paris. They extended over four weeks, during which period a portion of the ration of oats was replaced by the cocoa-nut meal, whilst for the sake of comparison, five other horses were dieted as usual. During the first two weeks the animals received but little exercise, being driven from 13 to 14 kilometres daily, but during the last two weeks this distance was increased to from 24 to 28 kilometres *per diem*. The results were in favour of the cocoa-nut diet, as the following figures shew:—

Date when weighed.	Horse fed upon experimental diet.	Horse dieted as usual.
Jan. 12 .....	437.0 kg.	452.0 kg.
" 31 .....	434 "	450.4 "
Feb. 12 .....	410.1 "	446.0 "

It is calculated that by the use of this meal a saving in the cost of a horse's keep might be effected to the extent of some 50 francs *per annum*.—W. D. B.

#### The Utilization of Spent Hops. Bied. Centr. 1884, 142.

A SUGGESTION has been made in the *Bierbrauer* (vol. 14, page 505) that in large breweries it would repay the trouble to set aside the spent hops for paper factories, since they yield a very suitable fibre. In smaller breweries it is recommended to take advantage of their property of undergoing a fermentation when stored in the moist condition, and so obtain a considerable heating effect which might be applied, in hot beds, in place of stable dung, or for purposes of artificial incubation.—W. D. B.

*Treating Solutions containing Compounds of Ammonia to obtain useful products therefrom.* A. McDougall, Penrith. Eng. Pat. 4001, Aug. 17th, 1883.

THIS is an extension of Pat. 202, Jan. 14th, 1882. The solutions therein mentioned, containing compounds of ammonia, are well mixed with superphosphate, or else with peat or other organic matter which has been reduced to fine particles and acidified. The resulting mass, which should have an acid reaction, is dried, and constitutes a valuable manure.—A. R. D.

## XV.—SUGAR, GUMS, STARCHES, ETC.

*The Reduction of Alkaline Copper Solution by Dextrose.* F. Urech. Berl. Ber. 17, 495.

IN this paper the author gives the results of a series of determinations of the quantity of cupric oxide reduced by dextrose, when the latter is added to an equivalent quantity of Fehling's solution, and allowed to act for the times stated. These numbers are compared with those deduced from the two equations—

$$(1) a = \frac{\log. \text{nat} \left( \frac{u_0}{u} \right)}{t} \text{ and } (2) a' = \frac{u_0 - u}{u_0 t}$$

(cf. *Ibid.* 14, 1363 and 16, 762.)

The results are given in the following tables, of which I. represents the duration in hours; II. the percentage of residual copper, calculated by means of (2), in which  $a' = 0.0001122$ ; III. the percentage of residual copper calculated by means of (1), where  $a = 0.011122$ ; IV. the arithmetical mean of II. and III.; V. the author's experimental numbers, the result of observations on the action at 12°·5 C.

I.	II.	III.	IV.	V.
3 ...	96·81 ...	96·70 ...	96·74 ...	97·98
7 ...	92·75 ...	92·47 ...	92·61 ...	93·36
13 ...	87·26 ...	86·46 ...	86·86 ...	86·36
21 ...	80·90 ...	79·01 ...	79·97 ...	77·98
31 ...	74·24 ...	70·57 ...	72·41 ...	70·03
51 ...	63·65 ...	56·46 ...	60·05 ...	59·45
79 ...	53·17 ...	41·25 ...	47·21 ...	45·96
123 ...	42·02 ...	25·11 ...	33·61 ...	32·82
195 ...	31·37 ...	11·26 ...	21·31 ...	20·83
291 ...	23·48 ...	3·84 ...	13·66 ...	12·77
411 ...	17·82 ...	0·99 ...	9·23 ...	9·21
564 ...	13·65 ...	0·18 ...	6·91 ...	8·00

The agreement of the numbers in IV. with those in V. is sufficient proof of the correctness of the author's views. The essential difference between equations (1) and (2) is that the former takes into account the action of an accelerating moment, which is indeed the action of the alkali upon the dextrose, the reduction of the cupric oxide being a consequence of this.—C. F. C.

*Treatment of Molasses by Baryta or Strontia, &c.* J. Imray, London. Communicated by H. Leplay, Paris. Eng. Pat. 4592. September 26, 1883.

THIS invention relates to the extraction of sugar from molasses, or other saccharine materials, by means of the hydrates of baryta or strontia of a lower degree of hydration than those hitherto used, and a method for obtaining and regenerating these reagents. A monohydrate of either of these alkaline earths is produced either by treatment of the natural carbonate, or the carbonate resulting from the treatment of saccharine juices, as follows:—The carbonate is heated in a retort, and at a high temperature subjected to the action of superheated steam. Carbonic anhydride is driven off, and the hydrate becoming fused is drawn off. The respective composition of the hydrates thus obtained is (1) Baryta 89·5, water 10·5; (2) Strontia 85, water 15. As a consequence of lower hydration, if used with molasses, solution in water must first be effected, but with beet juice this is not requisite. The process is then conducted in the usual way. The apparatus consists of a vertical retort heated by a furnace the flue from which circulates

around it, and discharging the heated gases into a flue circulating around a number of iron vessels in which the carbonates are heated prior to their introduction into the retort. The top of the retort is provided with a charging hole, the lower part forming a receptacle for the fused hydrate. Pipes for the conveyance of superheated steam are so provided that it can be introduced either through the mass of the material or over its surface. It is preferable to introduce the carbonates moulded into the form of pipes, which are placed on a grating midway in the retort. The patentee claims:—(1) The use of monohydrates of strontia or baryta for treating saccharine juices. (2) The converting the carbonates by the action of heat and superheated steam into monohydrates. (3) The construction and arrangement of the apparatus for the conversion of the carbonates as described.—C. C. H.

*Apparatus for Drying and Cooling Sugar.* C. A. Day, London. Communicated by G. M. Newhall, Philadelphia, U.S.A. Eng. Pat. 4699, October 3, 1883.

IN drying sugar, and materials of a like nature, a satisfactory result is not obtained where the entire drying operation is carried on in one machine, because the fine portions of the dried products are carried by the currents of air traversing the machine, and deposited upon the wet crystals at the inlet end of the apparatus. Not only is the drying thereby retarded, but the appearance of the crystals is spoiled. To avoid this result, the present invention proposes to carry out the operation in three distinct steps. (1) By a preliminary drying in a first machine, where the operation is not carried to such a point as to produce dust. (2) A continuation of the process in a second machine, into which the crystals enter in such a condition that dust will not adhere to them. (3) The completion of the whole operation by introduction of the dried crystals into a cooler, through which a current of dry air is forced. The apparatus performing the first portion of the process consists of a cylinder formed by a number of tubes placed side by side, and communicating at one end with a common head, into which steam is supplied, their other ends being provided with a cap. The tubes are connected firmly together, and their interior surface is lagged or lined with sheet metal; their exterior is provided with curbs and spur wheels for the rotation of the whole apparatus. The material introduced through a hopper into one end of the tubular cylinder traverses the interior of the steam-heated rotating apparatus, and is discharged through spaces into a shoot communicating with the second apparatus. This consists of a trough provided with a cover, a hollow steam-heated shaft and suitable stirrers. The material is pushed along the interior in the opposite direction to a circulating current of air, and is allowed to drop through suitable apertures into the cooler. This portion of the apparatus consists of an inclined rotating drum, provided in its interior with projecting ribs for the retention of the crystals during rotation, so as to fully expose them to a current of air forced through the apparatus. The three portions of the whole combination as described are placed on floors above each other, so as to make the operation continuous. The whole invention and apparatus is covered by sixteen different claims.—C. C. H.

## XVI.—BREWING, WINES, SPIRITS, Etc.

*Improvements in the Method of Preserving Beers or other Fermented Liquors in good condition.* R. G. Bell, Oxford. Eng. Pat. 4532. September 22, 1883. Provisional Protection.

THE improvements consist in injecting carbonic acid gas into the liquors in the usual manner, and afterwards subjecting them to an increased temperature, varying according to the liquor under treatment. When necessary the patentee makes use of antiseptics. The objects of the invention are the prevention of turbidity, sediment, and flatness. For injecting the gas into the liquor aerated-water machinery is used.—G. H. B.



## XVII.—PRESERVING FOOD, MEATS, Etc.

*Preserving Food and other animal or vegetable substances.* J. Y. Johnson, London. A communication from La Société Anonyme de Conservation alimentaire, Paris. Eng. Pat. 4698. October 2, 1883.

USE is made of the antiseptic properties possessed by certain paraffins or hydrocarbons variously known as petreoline, vaseline, or soft paraffin, in which vegetable or organic matter may be preserved from decay. Petreoline is found in the native state, but may be prepared in the following manner: When the light products of crude petroleum have been distilled, the resulting tar is deodorised and mixed with six parts of animal charcoal. After exposure for twenty-four hours to a temperature of about 50° C. the mixture is placed in a suitable apparatus and washed with boiling ether. Petreoline is dissolved out, and the solution can be distilled to recover the ether. The residue left in the retort constitutes the petreoline. It may be applied to the articles to be preserved by spreading over the surface or by immersion. G. H. B.

*An Improved Antiseptic.* W. R. Lake, London. Communicated by T. A. Breithaupt, Strasbourg. Eng. Pat. 5326, November 10, 1883.

THE improved compound is prepared as follows: Pure rectified glycerine is, with the aid of heat, saturated with powdered boric acid. The crystals, which separate out on cooling, are twice redissolved and recrystallised in a solution of equal parts of water and alcohol. The resulting crystals are white, tasteless, and odourless, possessing strong antiseptic properties. The substance is applied to the preservation of meat, &c., by dipping the pieces for a period of about 10 minutes in a solution containing three parts of the powder in 100 parts of water, the immersion being several times repeated and the meat allowed to dry after each immersion. Beverages may be preserved by the addition of 5 grammes of the same solution to each hectolitre of the beverage. The compound is also stated to be of great value in surgery for the purpose of dressing rapidly healed wounds. The claim is for the improved antiseptic substantially as described.—C. C. H.

## XVIII.—SANITARY CHEMISTRY, DISINFECTANTS, Etc.

*Preventing the Pollution of Rivers.* T. D. Harries, Aberystwith. Eng. Pat. 1547, Oct. 30, 1883. Provisional Protection only granted.

THIS invention aims at the prevention of river pollution from metalliferous mines, by collecting the water used in the mechanical treatment of the ores in a large reservoir, from whence, instead of being discharged into the river, after settlement it is pumped back into one or two supply reservoirs feeding the dressing floors. The same water is therefore continuously used over again, the loss by evaporation or leakage being the only fresh supply required. The method is applicable, the patentee states, to tanneries and other manufacturing industries.—C. C. H.

*Filters.* G. F. Marshall, Battersea. Eng. Pat. 5310, November 9, 1883.

THE object of this invention is to simplify the construction of the class of apparatus used in filtering a domestic supply of water, so as to allow the removal and cleansing of the filtering medium more readily than heretofore. The body of the filter is constructed in the usual way, and provided with an internal flange, upon which rests the carbonaceous block acting as the filtering media. An indiarubber jointing ring is inserted between the flange and the block, and the latter is securely fastened

in place by means of a screwed rod of porcelain passing through the block of carbon. The upper and lower surfaces of this block are made solid, so that the water passes diagonally through the mass being collected into a tube through which the holding-down bolt passes, and emptying into the lower part of the filter. The novelty claimed is (1) the construction of a filter as described, so that the filtering media can be readily removed; (2) forming parts of the filtering block solid or non-porous for the purpose set forth.—C. C. H.

*Treatment of Waste Materials to Obtain Useful Products and Motive Power therefrom.* G. Epstein, London. Eng. Pat. 5436, November 17, 1883.

THE refuse from houses, markets, &c., or any waste materials containing starch or bodies of a starchy nature are treated with water and sulphuric acid in a suitable vessel in which they are raised to a temperature of 170° F., and subsequently, after prolonged digestion, to the boiling temperature. The starch, &c., is converted into glucose. The mash is strained and cooled, run into a large closed fermenting vessel, and fermented. The carbonic acid given off is stored in a receiver, superheated, and used for driving the engine furnishing the mechanical power required in the various stages of the different processes. The fermented wash is used upon a fresh quantity of material, and the alcohol formed during the fermentation is distilled off during the heating and digestion of the second mash. The acid may, in certain cases, be dispensed with, and the conversion effected by heat and diastase or malted grain. The patentee claims (1) the improved process, as described, for the utilisation of the waste materials by the conversion of their starch into sugar; (2) obtaining therefrom the different products.—C. C. H.

## XIX.—PAPER, PASTEBOARD, Etc.

*Treating Peat to Obtain Peat Fibre.* Eng. Pat. 5206, Nov. 2, 1883. W. Brown, Jun., Cookstown, Co. Tyrone, and Henry Gregg, Belfast.

PEAT is first softened by saturation in water, and then passed into a trough (also supplied with water) in which it is torn and cut by revolving cylinders armed with teeth, or by any other suitable tearing apparatus. From here it passes to a beater, whence an elevator carries it to a revolving sieve. While passing over this screen the peat is treated with jets of water, and being now free from all extraneous matter requires simply to be dried to become a marketable product.—A. R. D.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

*Synthetical Piperidine.* By a Ladenburg and C. F. Roth. Berl. Ber. 17, 513.

THE authors have finally established the identity of the base obtained by the action of sodium upon pyridine in alcoholic solution. Prepared in large quantities its properties were found to be identical with those of piperidine obtained from pepper. Further, by the action of Carbon disulphide (Cahours: Ann. Chim. Phys. [3] 38, 88) both were

converted into the same compound  $\text{CS} \begin{cases} \text{N.C}_5\text{H}_{10} \\ \text{SH.NH.C}_5\text{H}_9 \end{cases}$

By the reaction in question the authors have succeeded in obtaining 70 per cent. of the theoretical yield of the synthetical base, the identity of which is thus established.—C. F. C.

## Obituary.

### HENRY WATTS, F.R.S.

THE black border on the cover of the July issue of the *Journal of the Chemical Society* and the empty space where for so many years we have been accustomed to see the familiar and highly respected name of the Editor of that Journal, HENRY WATTS, F.R.S., bore silent but pathetic testimony to the fact that that most conscientious and talented editor, that translator and compiler of world-wide fame, one of the most indefatigable and unwearied contributors not only to the advance and extension of the literature of pure Chemistry in this or any other country, but one of the first pioneers, and certainly the most effective one, in the advancement of a literature of chemistry applied to certain special branches of the Arts and Manufactures—had been suddenly removed from our midst.

Henry Watts was born in London on the 20th of January, 1815. He received his early education in a private school, and as a student attended lectures in the University College, London. In 1841 he graduated as Bachelor of Arts, in the University of London. In 1846, as assistant to the late Professor Fownes, he entered the Birkbeck Laboratory, and directed the work of the students till the death of his friend Fownes in 1849, and further continued his work in that Institution under Professor Williamson till 1857. Watts's first great effort as a translator and compiler was in 1848, when he was engaged by the Cavendish Society to prepare a translation with additions of the large German work of Leopold Gmelin, viz., Gmelin's "Handbuch der Chemie." The work thus produced extended to 18 volumes, and absorbed the greater portion of Mr. Watts's time for more than 20 years, the last volume and the index appearing in 1872. The Dictionary of Chemistry, commenced about the year 1858, at the request of the eminent publishers, Messrs. Longmans & Co., appeared in 1868 in five large octavo volumes. This work was kept up to date by the periodical issue of three supplementary volumes, one in 1872, the second in 1875, and a third, in two parts, in 1879 and 1881.

Mr. Watts also issued three editions of "Fownes's Manual of Chemistry," viz., the 10th in 1868, the 11th in 1872, and the 12th in 1877. For many years he held the appointments of editor of the *Journal of the Chemical Society* and of librarian to that Society. The writer, who served under Mr. Watts on the staff of abstractors for the *Journal of the Chemical Society* during eleven years, and was in frequent correspondence with him, feels sure that in bearing testimony to his extreme kindness and patience and unflinching courteousness, to which must be added an unswerving conscientiousness as editor, he is only expressing the views of many other chemists who have acted or are still acting on that staff.—W. S.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1884.

9219 H. H. Waddington, Manchester. Improvements in waterproof fabrics. June 20

9231 T. Macfarlane, Montreal, Canada. Improvements in the manufacture of chlorine and bleaching powder. June 20.  
 9243 C. D. Abel, London. Improvements in osmose apparatus. Communicated by H. Lepay, Paris. June 20  
 9252 E. Ostermayer, Ph.D., and M. Dittmar, Ph.D., Königsberg, Prussia. Improvements in the process of manufacturing chloroiodic bases. June 20  
 9251 C. J. Schofield, Clayton, Manchester. Improvements in or applicable to wicker hampers for carboys and other purposes. June 21  
 9263 T. Briggs and E. Webb, Salford. An improved method of dyeing and padding cloth, and in apparatus used therein. June 21  
 9269 A. T. D. Berrington, Pant-y-goitre, and John Parry, Ebbw Vale. Improvements in the methods of producing gases of high heating powers. June 21  
 9270 A. T. D. Berrington, Pant-y-goitre, and John Parry, Ebbw Vale, Monmouthshire. Improvements in the methods of utilising furnace gases. June 21  
 9273 Duke Fox, Dewsbury, Yorkshire. Improvements in the method of and apparatus for carbonising and drying woollen pieces, wool noils, waste, or rags. June 21  
 9285 R. Bell, Broxburn, N.B. Improvements in retorts or apparatus for distilling shales and the like. June 21  
 9301 W. Weldon, Burstow, Surrey. Improvements in urnaces for heating solid substances out of contact with the products of combustion of the fuel employed. Communicated by A. R. Pechiney et Cie., Salindres (Gard), France. June 23  
 9305 W. Weldon, Burstow, Surrey. Improvements in and relating to the manufacture of chlorine. Communicated by A. R. Pechiney et Cie., Salindres (Gard), France. June 23.  
 9306 W. Weldon, Burstow, Surrey. Improvements in obtaining partly in the free state and partly as vapour of hydrochloric acid the chlorine of the chloride of magnesium of aqueous solutions of chloride of magnesium, or of mixed solutions of chloride of magnesium and chloride of sodium. Partly communicated by A. R. Pechiney et Cie., Salindres (Gard), France. June 23  
 9307 W. Weldon, Burstow, Surrey. Improvements in the manufacture of chlorine and hydrochloric acid. Partly communicated by A. R. Pechiney et Cie., Salindres (Gard), France. June 23  
 9312 J. B. Thompson, New Cross, Kent. Improvements in bleaching. June 23  
 9317 W. Spence, London. Improvements in apparatus for the production of sulphuric acid. Communicated by J. J. Thyss, Bakou, Russia. June 23  
 9357 S. de la Grange Williams, Malvern, and J. A. B. Bennett, Llyndale, Worcestershire. Improvements in kilns for burning limestone, cement and bricks, and for other like purposes. June 24  
 9360 T. Dugard, London. A new and useful improvement in furnace fire door liners. Communicated by J. Mailer, San Francisco, U.S.A. June 24  
 9362 A. J. Boulton, London. Apparatus for concentrating sulphuric acid. Communicated by M. Willett, Newport, U.S.A. June 24  
 9371 G. A. Maples, London. An improved apparatus for preserving or otherwise treating milk, butter, fruit and other similar and perishable articles. June 24  
 9373 G. S. Jarman, Huddersfield, Yorkshire. An improved method or process of destroying vegetable substances contained in wool or woollen fabrics. June 24  
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The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

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James Taylor (late of Firs Hill Road), Osgathorpe Crescent, Sheffield.  
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*Local Sec.:* Thos. Tyrer, Garden Wharf, Church Road, Battersea, W.



CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE,  
Monday, June 9th, 1884.

## IRON AND ALUMINA AS CAUSES OF "REDUCTION" IN THE MANUFACTURE OF SUPERPHOSPHATE, &c.

BY H. B. YARDLEY.

THE main object of the superphosphate manufacturer is to obtain from the natural phosphatic materials at his disposal the maximum amount of soluble phosphoric acid, and to ensure its remaining in the soluble state, at any rate until he has got rid of it. With many, and indeed most, of the natural phosphates, this is the great difficulty.

A superphosphate is made, tested soon after making, and found to be everything that can be desired; it is stored for a month, and when again tested the soluble phosphoric acid, instead of being a trifle more in quantity, allowing for drying, is found to be more or less reduced.

This reduction, or "going back" of manures, has been ascribed principally to the presence of oxide of iron and alumina, and consequently phosphates containing large percentages of those constituents are at a discount in the market.

An experience of some years in the manufacture of manures in large bulks, and with almost every variety of phosphatic materials, led me to doubt the accuracy of the generally accepted impression that alumina is to be classed with the offending constituents, and accordingly I instituted the following experiments:—

1. To some clear watery extract of a superphosphate was added some solution of ferrous sulphate;

2. To another portion of similar extract was added some solution of alumina sulphate. In both solutions a whitish precipitate formed in a day or two. This was allowed to settle for a month, then collected in separate filters, washed, and examined. That from No. 1 was found to consist of a little calcium sulphate (washed out), and ferric phosphate  $\text{Fe}_2\text{O}_3\text{P}_2\text{O}_5$ . That from No. 2 was entirely calcium sulphate.

These results led me to make the further experiments tabulated:—

Experiments 4, 6, 8 and 10 were made for comparison with those containing iron and alumina.

In experiments 3, 4, and 5 the free acid is in very large excess; in 6, 7, 10, 11, and 12 it is in normal quantity, and in 8 and 9 it is deficient.

In experiments 3, 5, 7, and 11, where alumina is a constituent of the phosphatic mixture, and there is a sufficiency of free acid, the amount of  $\text{P}_2\text{O}_5$  insoluble in water shows no increase, even on keeping. In 9 there is a slight increase, but the free acid is very small in quantity, whilst in 12, where oxide of iron replaces alumina, the  $\text{P}_2\text{O}_5$  insoluble in water is largely increased, immediately as well as after keeping.

The foregoing experiments leave, I think, no doubt but that alumina is a harmless constituent of the natural phosphates, as far as the manufacture of superphosphates is concerned. I have purposely used the hydrate, as, although it has been stated that alumina exists always in the anhydrous condition in natural phosphates, I think that is open to question.

Since these experiments were made, this result has been verified on the large scale, in the use of a phosphate lately introduced, containing a considerable percentage—7 to 8—of alumina, with no more than 1 per cent. of iron oxide.

## THE DETERMINATION OF THE PERCENTAGE OF OIL IN PARAFFIN SCALE.

BY BOVERTON REDWOOD, F.I.C., F.C.S.

WHEN, some years ago, I was asked to commence the systematic examination of paraffin scale for contract purposes, it became necessary for me to devise a method of ascertaining the percentage of oil which should be capable of yielding practically unvarying results in the hands of different operators, or in successive tests by the same experimenter.

My first step was to obtain a convenient form of press, with an arrangement for indicating the amount of pressure applied. A hydraulic press naturally

MIXED.	Experiment	3	4	5	6	7	8	9	10	11	12
Grains pure Tricalcium Phosphate .....		200	200	200	200	200	200	200	175	175	200
„ „ Calcium Carbonate .....		25	25	25	25	25	25	25	50	25	25
„ „ Silica .....		12.5	25	12.5	25	12.5	25	12.5	25	25	12.5
„ „ Alumina Hydrate .....		12.5	—	12.5	—	12.5	—	12.5	—	25	—
„ „ Ferrie Oxide and Hydrate ..		—	—	—	—	—	—	—	—	—	12.5
		250	250	250	250	250	250	250	250	250	250
Grains Chamber Acid, 115°T .....		300	313	313	250	250	180	180	250	250	250
<i>Tested day after mixing gave—</i>											
$\text{P}_2\text{O}_5$ soluble in water .....		16.67	14.76	15.12	16.11	16.02	15.68	15.58	11.93	13.62	15.90
„ insoluble .....		0.43	0.17	0.17	0.40	0.33	5.42	6.89	0.81	0.40	1.71
Free Acid, calculated as $\text{SO}_3$ .....		15.95	8.67	8.67	3.91	3.91	0.48	0.18	undtd	undtd	4.06
<i>Re-tested after one month—</i>											
$\text{P}_2\text{O}_5$ soluble in water .....		15.82	14.71	14.59	16.81	15.11	14.85	13.72	11.19	14.83	15.82
„ insoluble .....		traces	0.20	0.23	0.51	0.48	7.78	9.16	0.58	0.35	2.43
Free Acid, calculated as $\text{SO}_3$ .....		15.82	8.57	8.18	3.91	3.75	0.77	0.48	undtd	undtd	2.58

suggested itself as well adapted for the purpose, in consequence of the facility with which the pressure applied by such a press can be measured; but from past experience, which may, however, have been unfortunate, I felt doubtful whether a *small* hydraulic press could be relied upon to work satisfactorily. I accordingly decided to adopt a modification of the "Boomer" press, wherein the minute deflection of the cross-head is magnified by levers, and thus conspicuously exhibited on a scale. After several failures, Messrs. John H. Ladd & Co. succeeded in producing a press with which I was satisfied. This apparatus consists of a steel cross-head, fixed above a substantial bed-plate and carrying a vertical screw, which terminates at the lower end in a solid plunger, about  $5\frac{1}{2}$  inches in diameter. Attached to the cross-head is the pressure gauge already referred to.

The sample of scale is placed in a shallow iron cup, between circular pieces of calico, cut to fit, and with a sufficient number of sheets of blotting paper of similar size, above and below, to absorb the oil. The plunger of the press, which accurately fits the cup, is then screwed down by means of a lever, about 4 feet in length, and as the pressure increases, the deflection of the steel cross-head actuates the pointer of the gauge through an intermediate lever, the amount being shown on a graduated scale. There are ten divisions of the scale, representing from one to ten tons pressure on the whole surface of the press-cake, equal to about 7 ewt. per square inch. Mercury cups are provided in the plunger and bed-plate of the press for accurately taking the temperature. The loss in weight sustained by the sample gives the percentage of oil.

It will be seen that the pressure is really applied through the medium of a very powerful spring, and this arrangement possesses the advantages of being independent of the influence of friction of the screw (which may vary considerably), and of being comparatively little affected by the consolidation of the material as the oil is expressed. Mr. McCutcheon (Young's Paraffin Light and Mineral Oil Company), has kindly furnished me with particulars of a paraffin scale testing press he has recently constructed, in which spiral springs are substituted for the flat spring of my press. If these spiral springs remain of unimpaired elasticity after prolonged use, I have no doubt that this press will furnish results quite as satisfactory as those with which my press yields.

Paraffin scale consisting, as is well known, of a mixture of hydrocarbons, ranging from the solid to the liquid, through all the intermediate stages, the determination of the percentage of oil must of necessity be an arbitrary test, depending upon the temperature at which the pressing takes place, the amount of pressure applied, and the length of time for which it is continued, as well as the quantity of material operated upon, in relation to the diameter of the press-cake. It is therefore necessary to decide upon these points by arrangement.

In a series of comparative experiments I found that, in the use of the press described, at a temperature of 60° Fahr., with an indicated pressure of nine tons, applied for five minutes, I obtained, with 500 grains of material, results agreeing with those yielded, on the average, under certain contract conditions which had been previously observed. and my tests of "hard scale" have accordingly been made on this basis.

The press, and method of working described, have now been in use in the United States, and in works in this country, as well as by myself, since 1881-82, and have furnished very satisfactory results. The accuracy of the process is fairly shown by the following figures the majority of which represent duplicate

experiments, made for business purposes at various times:—

Example.	Percentage of Oil.		Difference.
	1st Experiment.	2nd Experiment.	
1.....	6.68	6.64	0.04 per cent.
2.....	4.42	4.32	0.10 "
3.....	14.36	14.46	0.10 "
4.....	13.48	13.48	nil. "
5.....	4.51	4.52	0.01 "
6.....	11.81	11.76	0.05 "
7.....	14.08	14.06	0.02 "
8.....	3.36	3.44	0.08 "
9.....	7.16	6.94	0.22 "

Example 10.	1st experiment .....	1.460 per cent. of oil.
	2nd ..	1.458 "
	3rd ..	1.486 "
	4th ..	1.500 "
	5th ..	1.440 "

Maximum difference..... 0.06 per cent.

Example 11.	1st experiment .....	6.78 per cent. of oil.
	2nd ..	6.70 "
	3rd ..	6.70 "
	Maximum difference.....	0.08 per cent.

It is, no doubt, a theoretical defect in any form of spring press that the strength of the spring cannot, without a hydraulic gauge, be directly tested from time to time, but the use of a standard sample of scale affords a sufficiently accurate method of indirectly testing the spring, or comparing one press with another.

The experiments recorded below were made with a view of ascertaining the effect of variations of temperature, time, quantity, and pressure, and I have to thank my assistant, Mr. Moon, for the care he has bestowed upon them.

Number of Experiment.	Mark of Sample.	Quantity in Grains.	Pressure in Tons.	Temperature in Degrees F.	Time in Minutes.	Percentage of Oil.
1	A	500	9	60	5	5.3
2	"	"	"	"	15	7.52
3	"	"	"	"	30	7.92
4	"	"	"	"	60	8.62
5	"	"	"	"	120	8.58
6	"	"	"	65	5	7.78
7	"	"	"	"	15	9.76
8	"	"	"	"	30	9.72
9	"	"	"	70	5	10.82
10	"	"	"	"	15	11.62
11	"	"	"	"	30	12.16
12	"	"	5	60	30	6.76
13	"	250	9	"	5	6.4
14	"	"	"	"	15	8.16
15	"	"	"	"	30	8.6
16	"	"	"	"	60	9.08
17	"	"	"	"	120	9.04
18	"	"	"	65	5	8.6
19	"	"	"	"	15	9.2
20	"	"	"	"	30	9.52
21	"	"	"	70	5	11.52
22	"	"	"	"	15	12.0
23	"	"	"	"	30	12.56
24	"	"	5	60	30	7.81
25	B	500	9	60	5	11.04
26	"	"	"	"	15	12.22
27	"	"	"	"	30	13.08
28	"	"	"	"	60	12.98
29	"	"	"	"	120	13.22
30	"	"	"	65	5	12.74
31	"	"	"	"	15	13.48
32	"	"	"	"	30	13.72
33	"	"	"	70	5	14.16
34	"	"	"	"	15	14.72
35	"	"	"	"	30	15.2
36	"	"	5	60	30	12.74
37	"	250	9	"	5	11.68
38	"	"	"	"	15	12.88
39	"	"	"	"	30	13.24
40	"	"	"	"	60	13.20
41	"	"	"	"	120	13.6
42	"	"	"	65	5	13.2
43	"	"	"	"	15	13.6
44	"	"	"	"	30	14.0
45	"	"	"	70	5	14.64
46	"	"	"	"	15	15.08
47	"	"	"	"	30	15.32



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MANCHESTER TECHNICAL SCHOOL,

June 10th, 1884.

MR. IVAN LEVINSTEIN IN THE CHAIR.

### A NOTE ON THE EXTRACTION OF BENZENE FROM COAL GAS.

BY GEORGE E. DAVIS.

IN my paper which I read before the members of the Birmingham Section, "On the Distillation of Coal and Extraction of Benzene from the Gas," I endeavoured to give a short history of the processes so far as I was able. After the appearance of this paper in our valuable Journal my attention was called to the fact that I had made no mention of the late Mr. Cusiter's researches. I had already mentioned that in February, 1869, Caro obtained a patent for absorbing the benzol by means of heavy oils, but it now appears that Mr. Cusiter made some very interesting experiments in this direction as early as 1868, but it is not certain that they were made public until the results were read before the West of Scotland Association of Gas Managers by his brother-in-law, Mr. W. Young, of Paisley, in 1874. Mr. Tervet, whose name is well known to you, as the reader of a very interesting paper on "The Production of Ammonia from Coal," has furnished me with this paper, and some extracts from it may be interesting to you, especially as so much has been said lately of the temperature of carbonization. It appears that in the winter of 1868 Mr. Cusiter was experimenting with glycerine as a substitute for water in gas meters, and finding this fluid unsuitable for his purpose he tried mineral oils, and to his surprise, when the meter was charged with a mineral oil of 840 sp. gr., a 28 candle cannell gas was reduced to 14 candles luminosity. When the same gas was passed through paraffin spirit of 768 sp. gr. at a temperature of 50° F., the loss of illuminating power was five per cent; when the temperature was raised to 60° F. the result was nil; whilst when a temperature of 70° F. was employed, the illuminating power of the gas was actually increased some seven per cent. By passing the same gas through either of the heavy oils the loss of light was 65 per cent.; whilst by agitating the oil and gas together "the light-giving constituents were almost entirely removed." I mention these facts to show that Mr. Cusiter must have worked very assiduously at this subject, and his conclusions will no doubt be accepted as correct by those who have worked in this direction in later years. It is stated that gas made from coal at a low temperature, similar to that used for making paraffin oil, gave a distillate of 34 per cent. permanent gas and 66 per cent. of liquid hydro-

Number of Experiment.	Mark of Sample.	Quantity in Grains.	Pressure in Tons.	Temperature in Degrees F.	Time in Minutes.	Percentage of Oil.
48	C	250	5	60	30	12.76
49	"	500	9	60	5	6.12
50	"	"	"	"	15	7.81
51	"	"	"	"	30	8.60
52	"	"	"	65	5	8.18
53	"	"	"	"	15	8.22
54	"	"	"	"	30	9.6
55	"	"	"	70	5	10.11
56	"	"	"	"	15	10.1
57	"	"	"	"	30	10.22
58	"	"	"	75	5	10.66
59	"	"	5	60	30	10.9
60	"	250	9	"	5	7.3
61	"	"	"	"	15	6.84
62	"	"	"	"	30	8.41
63	"	"	"	65	5	8.8
64	"	"	"	"	15	8.92
65	"	"	"	"	30	10.2
66	"	"	"	70	5	10.2
67	"	"	"	"	15	10.52
68	"	"	"	"	30	10.64
69	"	"	"	75	5	10.76
70	"	"	5	60	20	11.68
71	D	500	9	60	5	8.1
72	"	"	"	55	"	10.31
73	"	"	"	50	"	6.36
74	"	"	"	60	"	4.52
75	E	"	"	55	"	10.26
76	"	"	"	50	"	6.0
77	F	"	"	60	"	4.4
78	"	"	"	55	"	9.0
79	"	"	"	50	"	8.0
80	G	750	"	60	"	6.54
81	"	500	"	"	"	1.9
82	"	250	5	"	"	1.2
						1.24

From an examination of the foregoing figures it will be seen that the results are most influenced by variations of temperature, and of the time during which the pressure is continued. It is, unfortunately, not possible to provide a table of corrections for temperature, as all samples are not equally affected by variations, and the most important conclusion to be drawn from the results recorded is, that scrupulous attention should be paid to the temperature of the press and of the sample, the specified temperature being rigidly observed.

At first sight it would appear that a longer period than five minutes should be allowed for the expression of the oil, but having regard (a) to the close agreement between the results obtained in this length of time, (b) to the increased difficulty of maintaining a given temperature for extended periods, (c) to the importance, if not necessity, occasionally, of being able to furnish results quickly, and (d) to the fact that the test must, under any circumstances, be only an arbitrary one, the value of which to the buyer lies in its association by experience, with results obtained in practically working the material, I am not prepared to admit that any necessity for extending the time has been shown by my experiments; moreover, I am of opinion that, in some cases at any rate, a portion of the semi-solid hydro-carbons contained in the scale, which certainly cannot fairly be called "oil," is gradually squeezed out during a prolonged pressure of so thin a cake.

I find that samples may be efficiently and expeditiously averaged by passing them through a small hand sausage machine, with fixed knives and revolving arms which can be removed for cleaning.

carbons, very similar in composition to light paraffin or petroleum spirit, having a sp. gr. of .706, and distilling

10% @ 128° F.  
50% @ 176° F.  
82% @ 212° F.

On the other hand, gas made from the same coal at a high temperature gave a distillate, having a sp. gr. of .852, very much resembling the most volatile portions of the distillate from ordinary coal tar, and consisted principally of benzenes, together with a number of other volatile hydrocarbons. It is most probable that in actual practice one series is never obtained in absolute purity. In distilling at high temperatures the aromatic series is slightly contaminated with paraffins, olefines and other hydrocarbons, which should be separated; while in distilling at low temperatures there are always some of the aromatic series intermingled with the paraffins and olefines.

I recently noticed that a patent had been taken out for the production of benzene by passing the vapour of petroleum through red-hot tubes. I have made a great many experiments on this subject, and am able to say that producers of benzene need not fear any rival in this direction. I have, however, obtained results interesting in another way, and may perhaps read a paper on the subject before the Section next session.

Before concluding I wish to say a few words relative to some remarks printed in a paper read by Mr. Watson Smith before the Liverpool Section. He there says (see p. 500, Vol. II. *Jour. Soc. Chem. Ind.*): "Benzol may be recovered from coal gases by means of refrigeration, pressure, and the use of some chemical reagents." Now M. Meunier, whose words are quoted, does not give the results of any of his own experiments, and Mr. Watson Smith's remarks are scarcely a correct representation of the result of the German chemists, to whom M. Meunier alludes (evidently Caro's patent of February 19, 1869). It would seem from the remarks made at Liverpool that refrigeration was combined with the use of pressure and chemical reagents. It was, therefore, important to ascertain the exact words used by M. Meunier in *Cosmos* (October 30, 1869), as I was not aware that anyone had combined the operation of refrigeration with that of absorption for the purpose of extracting benzene from coal gas. On referring to the original I find M. Meunier is very definite on the subject. He writes:—"La benzine employée actuellement dans le commerce a sa source principale dans le goudron obtenu par la distillation de la houille; cependant cette substance peut encore être obtenue par le refroidissement, ou la compression du gaz, ou bien par l'action de différents réactifs, l'acide azotique, l'acide sulfurique, le chlore, le brome, sur le gaz d'éclairage, et au rapport du *Journal de l'Eclairage au gaz* plusieurs chimistes allemands qui se sont préoccupés des moyens d'extraire la benzine entraînée par le gaz sont arrivés à un procédé d'une remarquable simplicité. Il résulte de leurs recherches que pour ne point modifier les propriétés chimiques des corps cherchés (la benzine et ses homologues), on doit de préférence mettre en jeu des matières qui peuvent dissoudre ces corps. Dans ce but ils adoptent toutes les huiles de goudron, notamment les huiles qui bouillent à une température plus élevée que le degré d'ébullition de la benzine. Le pétrole, les schistes, les huiles grasses, une foule de substances semblables, conviennent parfaitement. On met le gaz de houille en contact très intime avec les dissolvants que nous venons de citer; la séparation se fait ensuite par distillations fractionnées. La séparation terminée, on peut reprendre le produit et l'employer de nouveau pour dissoudre la benzine."

Such was a summary of what was known then, and by the light of recent experience we may be able to criticise the statements. It is very evident that Meunier's idea was that "benzine" might be obtained from lighting gas by cooling. Well, I have tried cooling, and my results have been very unsatisfactory. The ordinary Manchester gas may be cooled down many degrees below zero without depositing anything appreciable.

Compression yields slightly better results, for by compressing an 18 candle gas to 200lb. on the square inch I have been able to get about six ounces of a liquid consisting mainly of benzene from 1000 cubic feet of the gas. With regard to the process of dissolving out the "benzine" with heavy oils, I am afraid M. Meunier was but imperfectly acquainted with the process when he speaks about regaining the "benzine" by fractional distillation; such a process could not possibly yield all the benzene dissolved, and no mention is made of the details which ensure a good yield and so make such a process a commercial success.

I am happy to be able to inform the Section that the process for the extraction of benzenes from coal gas, and of utilising the remaining by-products as described by me before the Birmingham Section, will soon be actively at work at the Rockingham Gas Works, and I hope also to be a pioneer in the process of carrying on manufacturing industries without the emission of smoke or sulphurous acid. The gas which has been first deprived of sulphuretted hydrogen in the ordinary way, and finally of benzol and its homologues has, to use the words of the *Chemical News* of November 5, 1869, "lost so much of its illuminating power as to be only fit for heating purposes;" this will be used for heating the retorts, and by this means I hope to possess an advantage over all other coal consumers in that the products of combustion will not be in the slightest degree injurious to vegetation. What a change there would be in the aspect of the manufacturing districts of this country if the sulphur were extracted from all the coal used as fuel before actual combustion!

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## Journal and Patent Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*On a Means of Obviating an Inconvenience in the ordinary Wash Bottle.* C. Sölscher. Berl. Ber. 17, 1080.

It is a matter of common observation that in using a washing bottle the water issues at first with a jerk, so that there is frequently a danger of losing a portion of the precipitate or liquid which is being washed. The author proposes to prevent this by sealing on to the lower portion of the tube which dips under the water a similar tube, slightly enlarged at the junction into a double bulb, in which is placed a piece of glass rod drawn out at its lower end into a cone. The two tubes may be connected by a piece of india-rubber tubing. The cone acts as a valve, so that when air is forced into the flask, the cone rises and the water passes it, but when the operator ceases to blow the cone falls and closes the tube, leaving it quite full of water. When the bottle is again used, the tube is already full, and the water issues quietly at once.—S. Y.

*An Improved Composition for Preventing Incrustation in Steam Boilers.* Thomas Lowe, Radford, Notts. Eng. Pat. 4508, March 13, 1884.

A COMPOUND is formed, said to consist of 12 per cent. caustic soda, 8 per cent. charcoal, 2 per cent. Irish moss, 80 per cent. malt combs or dust, or the refuse from dried malt, and sufficient water to make it of the proper consistency. About 14lbs. per week per 50 horse-power boiler will generally be found to answer. The use of malt refuse is claimed.—J. T.

### II.—FUEL, GAS, AND LIGHT.

*Improvements in the Construction of Coke Ovens.* J. Jameson. Eng. Pat. 4766. Oct. 8, 1883.

THE inventor claims the use of a separate wall or casing to a coke oven, enclosing a space wherein the pressure of air outside the actual oven wall is made approximately equal to the pressure within the oven, so as to stop or diminish leakage of air into the charge in the oven. His invention may be so modified that the air leaking into the protecting case may be passed into the upper part of certain ovens of a range, and excluded from others by opening or closing apertures provided for the purpose, the main object being to prevent ingress of air into the oven at an undesired time and place.—D. B.

*Manufacture of Artificial Fuel.* E. Marriott, Highgate. Eng. Pat. 4771, October 8, 1883.

THIS invention relates to the manufacture of perforated blocks or bricks of patent fuel, composed of coal dust, slack, and similar waste material, agglomerated by admixture with bituminous and other substances in suitable proportions. The agglomerated materials are formed into bricks or blocks by compression in moulds of suitable shape and dimensions. The machine is of such a construction that the moulding and perforation of the blocks are performed at one and the same operation.—E. G. C.

*Apparatus for Cleaning, Separating and Classifying Coal, &c.* A. Sottiaux, Belgium. Eng. Pat. 4783, October 9, 1883.

ACCORDING to this invention, the impure or mixed coal, or other material, is caused to fall from a hopper, provided with a rotating feed cylinder carrying radial blades, through a case or chamber, the bottom of which is divided into compartments of suitable dimensions. At the upper part of the case, and immediately beneath the hopper, are several horizontal rows of bars, the spaces in one row alternating with the bars of the row next beneath. The case is open on one side, and communicates through the opening with an enclosed space serving to collect the dust. The other side of the case is provided with a series of tuyeres or nozzles, at different levels, furnished with valves controlled by suitable handles, and communicating with a vertical main or supply pipe through which air is sent under pressure. The coal placed in the hopper is caused to fall in a regulated stream from the feed cylinder on to the horizontal bars, which divide the coal in such a manner that fragments of schistose or slaty matter (which are generally of a flat shape), in falling through the case, present themselves edgewise before the nozzles, and are received in the compartment at the bottom of the case immediately beneath the hopper. The dust and lighter particles of coal are blown directly through the open side of the case into the enclosed space beyond, and the heavier portions of coal fall, in order according to their size and weight, into the compartments farthest from the nozzles. The inventor also describes another apparatus for the same purpose, consisting of a species of turbine rotating on a vertical axis in the centre of a case provided with a series of concentric cylinders. The turbine possesses a flat bottom in which are formed a number of radial channels rising towards the centre in a curve converging into a circular opening for the reception of the materials. The materials supplied at the centre of the apparatus enter the curved channels, and the heavier pieces are thrown by centrifugal action into annular spaces between the cylinders, which form so many compartments, in which the materials are collected in order of gravity.—E. G. C.

*Purifying Lighting Gas.* W. E. Gedge, London. (A Communication from Jean E. A. Servel, M.D., St. Etienne, France). Eng. Pat. 4844, Oct. 11, 1883.

IN this invention the action of the gas produces the circulation of the purifying liquids through which it

passes. The inventor uses four vats or vessels, diminishing in height, and communicating with each other at top and bottom, near the upper and lower surfaces of the liquids. Each vat contains an iron bell, the bottom of which forms an incline, and which is in communication, by the lower end of the incline, with the gas inlet or feed pipe. It is open at the top of the incline for the issue of liquid and gas. Slits or openings, made near this upper end, permit the purification of the volatile tar condensed by a guard placed above these slits, and parallel therewith. The apparatus may be composed of several series of vats, such as those above-mentioned, each of these series being in connection with a reservoir-vat, where the liquid circulates without being in contact with the gas. In these vats are placed the purifying liquids, which, according to this system, are as follows:—In the first series is utilised the ammoniacal water of gas regenerated by caustic lime, which is plunged, by the aid of a sack or bag, into the liquid in the reservoir-vats. The gas-water, thus regenerated, serves for the elimination of the ammonia, the carbonic acid, the sulphuretted hydrogen, and the carbon bisulphide. In the second series water alone is used to retain the excess of caustic ammonia. In all the vats the removal of the deleterious liquids takes place, free from atmospheric contact; further, all the purifying vats, properly so-called, receive the liquids from the reservoir-vats, and remain closed indefinitely. All danger of explosion is thus avoided; unhealthy exhalations cannot exist; and the usual manual labour disappears. This process of circulation of liquids may be utilised for the artificial carburation of gas. With this circulation of liquids moved by gas, the oxygen of the air may be retained, and the nitrogen eliminated. A valuable feed for high temperature furnaces will thus be provided.—E. G. C.

#### *Manufacture of Gas for Lighting and Heating Purposes.*

G. F. Redfern, Finsbury. (A Communication from S. C. Girandon, Paris). Eng. Pat. 4556, Oct. 12, 1883.

ACCORDING to this invention, air is carburetted by being forced into close vessels filled with "volatile oil or hydrocarbon," an air-pump being employed for the purpose. Several vessels or carburetters are employed, in the bottom of which open the pipes conveying the air: the mouths of these pipes are provided with nozzles or plates perforated with a number of holes, for the purpose of exposing large surfaces of air to the "volatile oil or hydrocarbon." The passage of the air is regulated by an air-regulator or bell placed between the air-pump and the carburetters. The oils, or hydrocarbons employed, may be produced from the distillation of naphtha, petroleum, mineral tar-oil, resin, etc.—E. G. C.

#### *Production of Ammonia and its compounds during the Process of making Coke or Gas.* H. Simon and Watson Smith. Eng. Pat. 4871, October 13, 1883.

THIS invention consists in injecting into gas retorts, coke ovens, or gas producers, provided with arrangements for recovering by-products, steam along with hydrocarbons in a liquid or gaseous form. Whilst the oxygen of the steam unites with the carbon, a portion of the hydrogen set free unites with the nitrogen of the partially decomposed coal forming ammonia, a portion also of the hydrogen unites with the sulphur of the fuel, so that the quality of the coke is improved by this treatment. The injection may be commenced preferably about the middle of the coking or gas-making processes, and the steam employed may be superheated, to prevent such cooling as might interfere with the distillation. The substances injected should be distributed uniformly over the incandescent fuel, which is best done by steam injectors, or regulated quantities of hydrocarbon may be dropped on heated surfaces, the vapour mixed with steam being distributed over the heated fuel. Atmospheric air must be excluded. The proportions of hydrocarbons to water (used as steam) are 58 to from 75 to 82 parts by weight; the supply (varying according to circumstances)

should, however, not exceed 580 pounds of hydrocarbons to from 750 to 820 pounds of water (steam) per ton of coal.—D. B.

#### *Obtaining Illuminating and Heating Gases and Useful Products from Coal and other matters.* Hartley Kenyon. Eng. Pat. 4951. October 17, 1883.

THE retorts have combustion chambers, and hollow walls or partitions, for the heating of the retorts and of air and steam. Coal rich in nitrogen, carbon, and hydrogen, and having a minimum of ash and iron is used. The retorts are worked in pairs. Illuminating gas having been driven off from a retort in a pair for a suitable time, the gas valve is closed, and highly superheated steam *per se* or mixed with "heated air" is discharged into the incandescent coke, and into the upper space superheated steam, "ammonia," and waste gas from the flues are discharged. Water-gas and "ammonium carbonate," are evolved, and are passed into an absorber containing "a solution of salt." The author of this invention prefers to use absorbers egg-shaped in section, and having in the lower part a revolving screw agitator and in the upper part a revolving agitator with arms which pass between partitions dipping into the solution. The gases in travelling from one compartment to the other become washed. "Sodium bicarbonate" is deposited and removed by the "screw." "Ammonium chloride" is formed, and passes into a second absorber charged with "soda waste or lime." From this absorber "ammonium sulphide" or "ammonia" escapes, calcium chloride and insoluble matter remaining behind. The "ammonium sulphide" is conveyed into "sulphurous acid," sulphur being precipitated and "ammonia" liberated. The latter is passed into the retorts to "absorb fresh quantities of carbonic anhydride." At a suitable stage of the process, when the second retort has been re-charged, the valve leading to the absorber is closed, and the gases are conveyed through the charge in the other retort to heat the same, the "ammonia" passing in with the superheated steam being in excess of the carbonic anhydride and hydrogen sulphide, whereby a gas free from these impurities is obtained. The gas passing through the hydrant is led into a washer and washed with ammonia liquor, "benzene being subsequently removed by suitable means." In some cases the author injects into the coke "zinc or iron chlorides," by means of the "superheated steam," in place of "ammonia."—D. B.

#### *Improvements in Apparatus used in the Manufacture of Gas.* J. Somerville, London. Eng. Pat. 4991. October 20, 1883.

THIS invention is applicable to apparatus used for different purposes in the manufacture of gas, but is especially adapted for use in connection with the dip-pipes and hydraulic main and in the construction of washers. With regard to dip-pipes the invention consists in either casting on or attaching to the lower part of the pipe a funnel-shaped cup, dipping into the liquid in the hydraulic, and having a hole at its bottom proportionate to the diameter of the dip-pipe, whilst for washers it consists in the insertion in each chamber of the washer of a number of short tubes beneath the lower part of each of which is arranged a funnel-shaped cup similar to the above. The action of the apparatus is the same in both cases, for as the gas passes down the dip or short tube the pressure it exerts on the surface of the liquid with which the hydraulic or washer is charged drives the liquid up the sides of the cup in the form of small bubbles or spray, so that the gas gets away readily from the dip and the short tube, whilst in the case of the washer, from the constant supply of liquid to the cups through the small orifices in the bottoms of them, the gas is efficiently washed. This addition to the dip-pipe greatly relieves the retort from pressure of gas when the coal is being distilled, and affords an effective seal to the dip-pipe when gas is not flowing through it, whilst applied to washers it ensures the more thorough washing of the gas and reduces the pressure such washing frequently produces.—D. B.



*Improvements in the Manufacture of Illuminating and Heating Gas and in Apparatus therefor.* A. J. Boulton, London. Eng. Pat. 5542. November 27, 1883. A communication from John Hanlon, New York, and J. E. Leadley, Camden, New Jersey.

THIS is a patent for the production of heating and illuminating gas by (1) the action of steam on incandescent fuel; (2) the distillation of bituminous or soft coal at such a temperature as to keep the resulting hydrocarbon vapours from destructive decomposition; (3) the vaporisation of liquid hydrocarbon; and (4) by combining the products of the above processes and converting them into a homogeneous fixed gas in a heated fixing chamber. Four different sets of apparatus for the working of the patent are described. The first consists mainly of a fire-brick iron-cased generating furnace having retorts situated in its upper portion; and another fire-brick iron-cased chamber filled with refractory material, and serving as the fixing chamber. The generator is supplied with fuel by moveable cylinders working on the top of the furnace, and provided with a water-chamber to prevent any distillation of the coal contained in them. There are also suitable arrangements for heating air and supplying steam, gas, and oil, as required. To work the apparatus, anthracite coal or coke is charged into the generating furnace, and an air blast admitted till the fuel is raised to incandescence, the hot gaseous products passing away to the fixing chamber, and after giving up their heat to the refractory material stored therein, escaping by a smoke stack. When all is suitably heated, the smoke stack is closed, the air blast turned off, steam is turned into the incandescent fuel (on the top of which some bituminous coal is now charged) and oil is admitted to the retorts at the top of the generating furnace. The hydrogen and carbonic oxide resulting from the decomposition of the steam rise through the fresh charge of coal now undergoing distillation, carry off the vapours disengaged therefrom, and then pass through the retorts, materially aiding in the vaporisation of the oil. The gaseous current now enters the heated fixing chamber, where it is converted into a fixed homogeneous gas, and whence it passes through a hydraulic seal box and stand pipe to the purifiers and storage-house. When the temperature falls below the point necessary for the decomposition of steam, the latter is turned off, the oil supply stopped, and the air blast once more admitted, as at first, to reheat the apparatus. Under the second head of the invention, a modified form of apparatus is described. Two fire-brick, iron-cased generating and decomposing furnaces have each in their lower portion a fuel chamber with a grate, and in the upper portion a steam superheating chamber, separated from the fuel chamber below by a perforated arch. The superheaters are partly filled with refractory brick-work, and above this with scrap iron. A vertical chute extends down through the superheater, so as to allow the supply of oil and coal to the fuel chambers. These latter are connected with one another by a pipe opening below the grate of each. From the superheaters pipes lead to the base of the fixing chamber, which is similar in principle to that already described. The whole system is raised to a suitable heat by the internal combustion of coal, an air blast being supplied both to and above the coal (the latter being to burn all carbonic oxide), and the resulting products of combustion, after passing through the fixing chamber, being allowed to escape until two beds of highly heated fuel about two feet thick are formed. If necessary, the fixing chamber may be heated by taking the generator gases into it before combustion, and there supplying an air blast. The operation of gas producing is carried on as follows: Generator No. 1 having been raised to a considerably higher temperature than No. 2, air is turned off and steam admitted into the superheater of the former, while a charge of bituminous coal and also a small quantity of oil is dropped through the chute of the latter. The hydrogen and undecomposed steam have then to pass downwards through the highly heated fuel in grate No. 1, along the connecting pipe and up through grate No. 2. Here the recently added charge of coal is distilling, and the volatile products are carried away by the upward current of gas from the other generator,

which now emerges from the top of the superheater into the fixing chamber, where it is converted into a homogeneous illuminating gas of high candle power. Of course, during this operation all connection between superheater No. 1 and the fixing chamber is cut off. When the temperature falls too low for the success of the operation the chambers are heated up again as at first—the generator which was before used for decomposing steam serving this time for the distillation of the bituminous coal, and *vice versa*. The carbonic oxide which passes through the now oxidised scrap iron in the process of heating up reduces it to metallic iron again. Under the third head is described a form of apparatus very similar to the last but simpler and better adapted for smaller works. One generator is used, having above it a smaller chamber carried by a perforated brick arch in which is contained coal to be distilled. The gases pass first into a mixing chamber provided with baffle plates upon which oil may be vaporised (if a high illuminating power is required), and thence to a fixing chamber in which it passes upwards through a long spiral channel round an internal cylindrical passage which serves for its outlet downwards. Steam, being passed into the incandescent fuel of the generator, is decomposed, and the resulting gases rising upwards carry off the vapours from the coal distilling above. The generator is fed by two chutes passing down on each side of the distilling chamber. The modifications described under the fourth head refer to a peculiarly constructed vaporising and mixing chamber, a peculiar fixing chamber, and a connected steam boiler. The first is a vertical cylinder with a trumpet-shaped mouth filled with double cones formed of bars or slats of brickwork. The second is another vertical cylinder, with a spiral flange winding round an inner hollow smaller cylinder. The gases pass downwards through the first chamber (which is provided at the top with a conical oil distributor), upwards round the spiral flange and downwards through the inner passage of the second, and finally away through the flues of a tubular boiler, their heat being utilised for the generation of steam.—A. R. D.

*Improvements in the Manufacture of Gas and in Apparatus therefor.* H. J. Rogers, Watford. Eng. Pat. 1204. January 11, 1884.

ACCORDING to one of the methods the inventor arranges coils of pipes, placed in any suitable position in the apparatus, and surrounds them with fire-bricks, forming a hot chamber, and beneath this chamber he fixes a mixing box for gas and air, the air being drawn in by means of the pressure of gas, and forming therewith an intense heating flame, by which means the coils are made red hot. A boiler filled with water is placed above or at the sides of the coils, or may overlap the coils, and form part of the hot chamber. The steam from this boiler, after superheating it by passing it through the hot chamber, is then conducted to an injector in the centre of the upper coil of pipes, the injector communicating with a tank containing crude or refined petroleum, shale, or coal oil, or other hydrocarbon oil. The oil is then injected into the coil, where it is thoroughly disintegrated by the steam and vaporised, a gas of a very high illuminating power being obtained. To render the apparatus more automatic, a tappet and lever arrangement is adopted, by means of which the various cocks may be opened or shut off. According to another arrangement a double retort, one placed inside the other, is used, the oil and steam passing first to the inner retort and then to the annular space formed between the two retorts, in which the materials are converted into a permanent gas. By this means the manufacture of gas is said to be simplified and cheapened, whilst the generating and storing apparatus is adapted for villas and small mansions, as well as for towns, villages, factories, ships, and large establishments.—D. B.

*Apparatus for Purifying and Clarifying Coal Gas.*  
F. A. Walker, Milton, near Gravesend. Eng. Pat.  
3886. February 25, 1884.

A SMALL vessel, made of galvanised iron, or any other suitable material, and filled with charcoal or coke, is interposed between the meter and the burner—the nearer the meter the better.—E. G. C.

*Improvements in Gas Burners for Consuming a Mixture of Gas and Air for Illuminating and Heating Purposes.* J. Lewis, Brockley. Eng. Pat. 3984. February 26, 1884.

ACCORDING to the specification of letters patent dated January 8, 1883, No. 195, induced currents of air were introduced to the pipe or chamber below the platinum wire gauze burner, to mix with gas under pressure before reaching the burner. According to the present improvements the induced currents of air are arranged so as to effect the combustion of the gas in the burner at its normal, or even below its normal pressure from the supply pipes. For this purpose, the platinum wire gauze burner is mounted upon a cone-shaped inlet passage over the gas supply pipe, the bottom of the cone being open to the atmosphere. The cone is surrounded by a chimney, an annular space being left between it and the bottom of the cone for the admission of air. Perforated plates are also arranged for the admission of air to the inlet cone and burner, below the cone and around the gas supply pipe; the openings can be closed, or partially closed, to regulate the admission of air to the burner.—E. G. C.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Liquid Hydrocarbons obtained by the Compression of Petroleum Gas.* By C. Greville Williams, F.R.S. Chem. News. 49. [1275]. 197.

THE author has found that when the gaseous hydrocarbons produced by subjecting petroleum to a high temperature, are compressed into cylinders, as in the Pintsch system, so much used for the illumination of railway carriages, a volatile liquid is condensed which contains benzene, toluene, and certain olefines. He has been engaged in the examination of this mixture of hydrocarbons since October, 1882, and desires to make public now what he has discovered up to the present time.

In order to separate the olefines from the benzenes, the hydrocarbon vapours were passed through a powerful rectifying column. The portions which distil over below 66°, were put aside for the preparation of the olefines required for conversion into hydriodates, in order to continue the work begun in 1862 (*Journ. Chem. Soc.*, xv. 359). Those portions distilling above 66° are used for the preparation of benzene and toluene. The olefines can be separated from the latter by agitating the hydrocarbons with a cold solution of potassium permanganate as demonstrated by Berthelot; or, better, by distilling from dilute nitric acid. By this last method nitro-compounds are produced, and these are being examined by the author. The nitric acid process, properly applied, gives good results; the amounts of benzene and toluene obtained in repeated experiments being within one per cent. of each other.

The specimens of the hydrocarbon from the various stations employing the Pintsch process differ considerably as to the amount of benzene and toluene they contain.

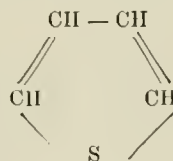
Seven specimens with which the author was favoured by Mr. Rickman managing director of the Pintsch Company, yielded the following numbers:—

Specimen.	Specific Gravity.	Percentage of Benzene and Toluene.
A .....	0.850	65.6
B .....	0.835	54.2
C .....	0.840	52.0
D .....	0.830	45.2
E .....	0.840	44.1
F .....	0.800	37.8
G .....	0.760	24.6

W. S.

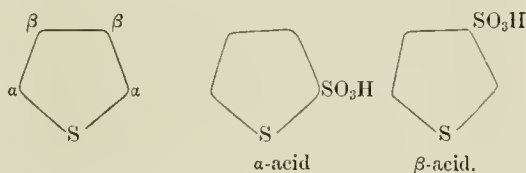
*Researches on Isomerism in the Thiophene Series.* V. Meyer. Ber. 17 [11] 1563.

FROM the constitutional formula proposed by the author for thiophene, viz.:—



it follows that, although this substance is so similar in its properties to benzene, yet it must differ from the latter in one point: the mono-substitution-products of thiophene must exist in two isomeric modifications. As yet no experimental proof of the constitution of thiophene has been given; and hence it appeared important to try whether the above conclusion would be verified by experiment. The following method was adopted:—Dibromthiophene,  $\text{C}_4\text{H}_2\text{Br}_2\text{S}$  was converted into a sulphonic acid,  $\text{C}_4\text{HBr}_2\text{S} - \text{SO}_3\text{H}$ , which was reduced by sodium-amalgam; the thiophene sulphonic acid thus obtained was compared with that obtained by the direct sulphonation of thiophene.

At Professor Meyer's suggestion, Hr. Langer undertook the investigation, and showed that the above sulphonic acids are different, the former being probably  $\beta$ -thiophenesulphonic acid, whilst the compound obtained by direct sulphonation is the  $\alpha$ -acid:—



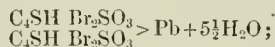
This is in entire accordance with the theoretical predictions, although, of course, it affords no absolute proof of the correctness of Victor Meyer's thiophene formula.

—D. E. J.

*On Isomeric Sulphonic Acids of Thiophene.* Joseph Langer. Ber. 17 [11] 1566.

THE author describes the methods adopted in preparing the compounds mentioned in V. Meyer's paper.

*Dibromthiophenemonosulphonic Acid*,  $\text{C}_4\text{HBr}_2\text{S} - \text{SO}_3\text{H}$ .—Pure dibromthiophene (B.P. 205°–207°) is decomposed by an equal volume of melted pyrosulphuric acid. The liquid becomes deep red in colour; after standing for a few minutes it is poured into water, filtered, and converted in the usual manner into the lead-salt. The yield is very good. The lead-salt has the formula—



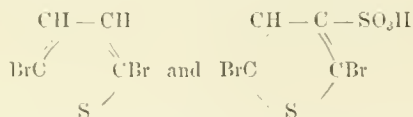
it crystallises from water in small, white, glittering crystals.

*Reduction of the Sulphonic Acid.*—The lead-salt is dissolved in water, the calculated quantity of caustic soda added, and the filtrate concentrated by evaporation. The sodium-salt is very easily reduced by 5 per cent. sodium-amalgam, which is gradually added until no further action takes place. It is remarkable that dibromthiophene itself is attacked only with great difficulty by nascent hydrogen, whereas its sulphonic acid is easily reduced; the same holds good for aromatic compounds (e.g. the bromobenzenes with their sulphonic and carbonic acids). By the action of  $\text{PCl}_5$  this sulphonic acid was converted into thiophene-sulphochloride,  $\text{C}_4\text{H}_2\text{S} - \text{SO}_2\text{Cl}$ , which forms large, colourless crystals; the acid prepared by direct sulphonation of thiophene yields a liquid chloride.

The position of the bromine-atoms in dibromthiophene is not yet known, but from analogy (e.g. paradibromben-



zene) we should expect that this body and dibromthiophenesulphonic acid have the formulæ—



The corresponding sulphonic acid would thus be a  $\beta$ -compound [I.], whereas the common acid would be an  $\alpha$ -compound [II.]



Further investigation will show whether these views are correct.—D. E. J.

*Improvements in Apparatus used in Connection with the Distillation of Tar or Oil and Treatment of Pitch.* F. Lennard, Shoreham. Eng. Pat. 4547. Sept. 24, 1883.

THE distillation of tar or oil having taken place, the inventor runs the residual hot pitch into closed wrought iron tanks or cylinders fitted internally with a number of wrought iron pipes supported by iron bars. Cold tar or oil is then pumped or forced through the pipes and becomes heated by the surrounding pitch, thereby adapting it for immediate distillation, whilst the cold tar in passing through the pipes lowers the temperature of the pitch and permits its removal much more speedily than is the present practice. Instead of the system of pipes a tank is used which is divided by a horizontal partition or false bottom so as to form two chambers, the lower chamber being divided by partitions stretching from top to bottom, and from one side to near the opposite side alternately, thereby forming a number of chambers each of which communicates with the adjoining one. A series of pipes of large diameter may also be used which are laid in the tank somewhat similarly to the above described and arranged to receive the hot pitch, such pipes being surrounded by the cold tar or oil.—D. B.

*Improvements in Treating Carbonaceous, Bituminous and other Substances, and in the Apparatus or means to be employed therefor.* H. Aitken. Eng. Pat. 4644. Sept. 29, 1883.

CERTAIN kinds of coal, shale and lignite and their analogues contain large amounts of water, which water on being exposed to high temperatures is driven off and simultaneously bursts or blows out the coal and so makes a very open light coke, which is carried off unburnt. The patentee dries the coal in a tower and blows or draws heated air through the same, or passes the products of combustion of a fire through the coal. The drying may, however, be effected in various ways. By this treatment a much denser and more serviceable coal for many purposes is obtained which produces a closer and heavier coke. It is also stated that such coal gives a higher yield of gas and that the benzene products are increased.

In treating coal for the production of coke, gas, oil, tar, bitumen, ammonia, etc., flues or spaces are placed in the bottoms of ordinary bee-hive ovens covered with perforated bricks. The flues are connected to a pipe communicating with condensers, scrubbers, and absorbers, having exhausters attached thereto for drawing the gases down through the coal as they are distilled. In the top portion of the oven a series of air holes is placed, and below or between the latter passages for admitting the gas. This mode of working gives oily products, but on putting one charge on top of another and drawing the products of distillation downwards, the gases passing through the hot coke become altered in their character to more of a tarry nature. In order to extract as much ammonia as

possible, water or steam is introduced in small quantities, when the coking is completed. The arrangement of ovens differs when it is desired to make a dense coke. An oven of another type is used for the production of soft or smithy coke, the retorts or chambers being of less depth and having more bottom surface. For the production of gas for illuminating purposes and the recovery of liquid products, brick chambers with thick walls are constructed, in which gas is burnt, the heat being raised to a very high degree, after which coal is introduced from the top. Metal may be used in place of brick for the division-walls. The heat of slag or iron as they come from the blast furnace is also utilised by covering the same with a cover preferably of metal lined with brick, and running therein the coal. There being a chance of explosion if the current of the gases coming into the oven is arrested by the stoppage of the exhauster, a valve is put on to the engine of the exhauster which is actuated by the exhauster, so that the moment the engine stops a steam valve is opened which admits steam both into the pipes and condensers, and into the coking chambers, whereby all risk of explosion is avoided.—D. B.

*Obtaining Volatile Hydrocarbons from Coal-gas.* E. Drew, London. Eng. Pat. 5039. October 23, 1883. Void.

IN the separation of volatile hydrocarbons from coal-gas it has been the practice to pass the gas through a heavy hydrocarbon oil contained in a series of vessels in such a manner that in drawing off the liquid from the first vessel of the series the entire series has for the time being to be put out of operation, as the liquid contained in the second vessel is run into the first, that in the third into the second, and so on through the entire series. To overcome these inconveniences and to keep the rest of the series of vessels constantly at work during the operation of emptying one of the vessels, the author of this invention arranges the series of vessels all on the same level and connects them together in a series, and to a main gas supply pipe and to an exhaust pipe by suitable pipes and cocks, so that any one or more of the vessels in the series may be cut off from the others whilst the latter can still be worked in a series. He also dispenses with the use of the intermediate vessel into which the hydrocarbon oil at the end of the operation is run, and from which it is raised into a tank by manual labour or mechanical means, by forcing the oil directly from the still into the tank by the pressure of the steam used for the distillation.—D. B.

*Manufacturing Light Essences by the Distillation of Coal, Wood, Turf, Oil, and similar Substances.* N. A. Hélonis, Paris. Eng. Pat. 5142. October 30, 1883. Provisional protection only.

THE author's process is based on the rational and permanent production of acetylene, a production which is favoured throughout the entire distillation of coal. This result is obtained by the intervention of three influences acting simultaneously:—1. A fixed temperature never exceeding dull red in the retorts. 2. The permanent action of a current of gas and steam previously superheated and led into the retorts during the whole time of the distillation. 3. The influence of a mean pressure of a fifth of an atmosphere maintained in the retorts by means of a pressure regulator arranged at the series of barrels. The three influences which constitute and characterise this invention have for result:—1. To produce the permanent formation of acetylene (generator of benzene). 2. To preserve the benzene from more complex combinations having for result the production of condensed carburets, which constitute heavy oils of less commercial value.—D. B.

*Bleaching and Purification of Petroleum and other Distillable Oils.* R. Baynes and J. Fearnside, Liverpool. Eng. Pat. 5208, November 2, 1883.

IN order to remove the dark colour possessed by mineral and other oils of a distillable nature, such as rosin oil,

the inventors take powdered charcoal or other absorbent not liable to be decomposed, dissolved or altered in composition by the oil, and mix it with the crude oil. The mixture is then distilled, whereby nearly all the colouring matter remains behind in the powder. The latter can be used for a fresh operation. As absorbents wood charcoal, coke, bone ash, fullers' earth, china clay, alumina earths (burnt and unburnt) have been used, but ordinary metallurgical coke or wood charcoal, steeped in a saturated solution of zinc chloride and well dried, was found to act best of any of these materials. It is important, however, that the material employed be absolutely dry to have the desired effect. The object of impregnating the charcoal with zinc chloride is to deprive the oil at the same time of its sulphur compounds and similar substances.—D. B.

*Process for the Separation of Benzene and its Homologues from the Liquid obtained by Compression of Petroleum Gas.* C. H. Greville Williams. Eng. Pat. 4663, 10th March, 1884.

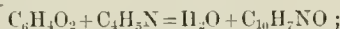
WHEN illuminating gas prepared from petroleum is compressed for the purpose of storage a liquid is obtained, containing homologues of olefiant gas, benzene and homologues of the latter. In order to separate the benzene, the liquid is subjected to fractional distillation, and separated into three portions. The first portion contains all that distils below 65°·5 and is rejected. The second portion contains all that distils above 65°·5 and below 87°·5, and the third contains all that distils above 87°·5. For the separation of the benzene the liquid is subjected to the action of dilute nitric acid (100 galls. require about 50 galls. commercial nitric acid and 50 galls. water), or of a mixture of an alkaline nitrate with sulphuric acid (100 galls. require about 190lb. commercial sodium nitrate and 218lb. of commercial oil of vitriol diluted with about 24½ galls. water). The mixture is then distilled preferably in a salt-glazed stoneware still, heated in a steam or water bath, or in a current of steam. The benzene and its homologues readily come over, the homologues of olefiant gas being converted into compounds, which remain behind in the still.—D. B.

#### IV.—COLOURING MATTERS AND DYES.

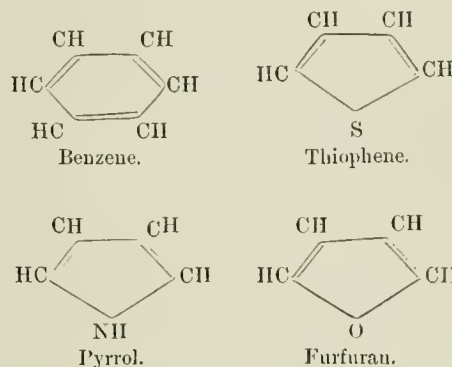
*Note on the Pyrrol Colouring Matters.* Victor Meyer and Otto Stadler. Berl. Ber. 17, 1034.

THE similarity of the colouring matters obtained by the action of pyrrol on certain ketones to the thiophene colouring matters has already been noticed (V. Meyer, Berl. Ber. 16, 2974, and Ciamician and Silber, 17, 142). When a cold aqueous solution of isatin is treated with pyrrol and a little dilute sulphuric acid a heavy blue precipitate, resembling indigo, is obtained. When both reagents, according to Ciamician and Silber, are dissolved in glacial acetic acid and boiled a deep blue solution is obtained, which apparently contains the same colouring matter. The substance could not be crystallised, and Victor Meyer did not propose any formula, while Ciamician and Silber suggested as a possibility the formula  $C_{23}H_{18}N_4O_3$ , with which their carbon and hydrogen determinations agreed. The authors, however, found a much lower percentage of nitrogen than that required by the above formula, and consider that the reaction is not so simple as would be inferred from it. The great resemblance of isatin to certain double ketones induced the authors to investigate the action of pyrrol on quinones. When a solution of phenanthrene quinone in acetic acid is treated with pyrrol and a little dilute sulphuric acid a brown precipitate is formed, which dissolves in chloroform with a beautiful red-violet colour. A solution of benzo-quinone gives a violet colouring matter with pyrrol, even without the addition of an acid. It dissolves in water and may be extracted with ether or precipitated with common salt, but is easily decomposed. Isatin and pyrrol also act on each other

slowly in absence of an acid; but the product is not a colouring matter. When an aqueous solution of benzo-quinone is treated with pyrrol and dilute sulphuric acid a dark-green precipitate is formed, which is insoluble in ether. It contains 8·6 per cent. of nitrogen, and might be supposed to be formed thus—



but the reaction is in reality not nearly so simple, for the filtrate from the colouring matter contains a considerable quantity of hydro-quinone, which may be extracted with ether. In the same way, when the filtrate from the colouring matter formed by the action of isatin on pyrrol is extracted with ether, colourless crystals are obtained in small quantity; and hence, in this case also, the reaction is not a simple one. Of great interest is the fact observed by Henninger (Soc. Chim., Paris, 22nd February, 1884), that furfuran resembles benzene and thiophene in its behaviour with bromine, while it exhibits a similarity to thiophene and pyrrol in its reactions with isatin and phenanthrene-quinone. A further proof of the parallelism, as shown below, between these substances is thus afforded—



—S. Y.

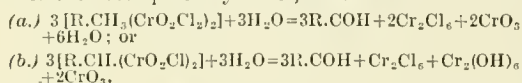
*On the Employment of Anhydrous Oxalic Acid in the Formation of Condensation Products.* R. Anschütz. Berl. Ber. 17, 1078.

CRYSTALLISED oxalic acid loses its two molecules of water slowly in the desiccator and quickly at 100°. The anhydrous acid is very hygroscopic, and, according to Löwig, acts as a dehydrating agent in the formation of oxalic ethers. The authors find a similar action in the formation of condensation products, even at temperatures above 100°. (1.) A solution of 5grms. benzaldehyde in 11·5grms. dimethylaniline was heated in a test tube in a paraffin bath to 110° for two hours without any action taking place. The addition of 7·5grms. powdered anhydrous oxalic acid under the same conditions brought about the formation of leucomalachite green, the yield being almost quantitative. (2.) A mixture of 6grms. diphenylamine, 3grms. benzaldehyde and 3grms. anhydrous oxalic acid was heated to 105° for 1½ hour. The product was treated with steam, boiled with water and then ammonia, and dissolved in benzene. The hydrochloride of the condensation product was precipitated with dry hydrochloric acid gas as a white powder, which turned green on exposure to the air. (3.) Benzaldehyde, benzylidiphenylamine and anhydrous oxalic acid gave the theoretical yield of a new condensation product, which does not appear to possess basic properties. (4.) A mixture of 7grms. resorcinol, 5grms. phthalic anhydride, and 35grms. of oxalic acid, when heated from 110° to 117° for ten hours, yielded 2·3grms. of fluorescein. Thus anhydrous oxalic acid acts as a dehydrating agent at a temperature at which it loses the whole of its water of crystallisation. The water does not, of course, remain combined with the oxalic acid, but escapes, and in the first experiment it was seen to condense on the cold parts of the tube. The whole of the oxalic acid may be recovered.—S. Y.

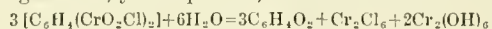


*On the Preparation of Aromatic Aldehydes according to Etard's reaction.* E. Bornemann. Ber. 17 [11], 1462.

MANY methods can be employed for converting the methyl groups in aromatic hydrocarbons into aldehyde-groups. One or two atoms of hydrogen in a methyl group can be replaced by chlorine; if the compound thus obtained is of the general formula  $R \cdot CHCl_2$ , like benzal-chloride (benzylidene-chloride), it is converted into an aldehyde  $R \cdot COH$  on boiling with lead nitrate; bodies of the formula  $R \cdot CH_2Cl$  are boiled with milk of lime under pressure. It is well known that large quantities of benzaldehyde are prepared from toluene according to the former method. In working on the laboratory scale, it is often very difficult to separate the above-mentioned bodies from the other chlorination-products of aromatic hydrocarbons. A. Etard has pointed out the following method of preparing aromatic aldehydes from small quantities of the corresponding hydrocarbons. Under suitable circumstances these hydrocarbons unite with chromyl chloride to form characteristic compounds, which are decomposed by water, thus—



If, however, no methyl-groups are present in the hydrocarbon, bodies similar to ketones are produced. Thus, for example, the compound obtained by the union of benzene with chromyl chloride (two molecules of HCl being set free) yields quinone, as follows—



*Preparation of the Toluic aldehydes.* — 1. *Meta-toluic aldehyde*  $C_6H_4 \cdot CH_3 \cdot COH$  (1, 3). 100 parts (2 molecules) of chromyl chloride, and 35 parts (about 1 mol.) of pure metaxylene are diluted with carbon bisulphide in the proportion of 15 : 100, and a solution of chromyl chloride is gradually added, the mixture being kept below  $47^\circ$  by cold water. After each addition of  $CrO_2Cl_2$ , it is necessary to wait until the red colour of the fluid has turned to chocolate-brown, otherwise unpleasant explosions may occur. Allow the brown crystalline precipitate of  $C_6H_4(CH_3)_2(CrO_2Cl_2)_2$  to settle down for twelve hours, then filter off over glass-wool and wash carefully with  $CS_2$ . The preparation is extremely hygroscopic, and must be freed from  $CS_2$  as follows. It is quickly placed in a flask provided with a well-fitting cork and a leading-tube; this is gently warmed on the water bath for 45 minutes, a current of dry air being pumped through meanwhile. The dry compound is thrown into water in small portions at a time, and decomposes—according to equation (a)—into meta-toluic aldehyde, chromic chloride and chromic acid. From this mixture the aldehyde should be at once extracted with ether, so as to avoid oxidation. The aldehyde is purified in the usual manner by converting it into the bisulphite-compound, decomposing this with sodium carbonate, and distilling over with steam. It is thus obtained as a colourless, highly-refractive oil, boiling at  $198^\circ$  to  $199^\circ$ , and smelling like bitter almonds.

II. *Orthotoluic aldehyde*  $C_6H_4 \cdot CH_3 \cdot CHO$  (1, 2) is obtained from orthoxylene by exactly the same method. It smells like benzaldehyde, and boils at  $199^\circ$  to  $200^\circ$ .

III. *Paratoluic aldehyde*  $C_6H_4 \cdot CH_3 \cdot CHO$  (1, 4) is also prepared in the same way from paraxylene. It smells like pepper, and boils at  $204^\circ$  to  $205^\circ$ .

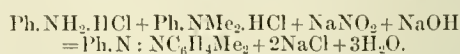
IV. Nitro-derivatives of meta-toluic aldehyde. *Ortho-nitrometa-toluic aldehyde*,  $C_6H_3 \cdot CH_3 \cdot NO_2 \cdot COH$  (1, 2, 3), is prepared by dropping one mol. m.-toluic aldehyde into somewhat more than one mol.  $KNO_3$  in conc.  $H_2SO_4$  (cold), pouring the solution into cold water and shaking up the product with sodium bisulphite. The yellow crystalline double-compound is decomposed with caustic soda, and the aldehyde is distilled off with steam. By the action of acetone and dilute alkalis it is easily and smoothly converted into m.-methyl-indigo. A di-nitro-derivative is obtained when somewhat more than two mols.  $KNO_3$  are employed.

*Meta-methylcinnamic Acid*,  $C_6H_4 \cdot CH_3 \cdot (CH=CH-COOH)$  (1, 3). This is obtained as a dirty white mass

by boiling together four parts of m.-toluic aldehyde, three of dry sodium acetate, and 10 of acetic anhydride, pouring into water acidulated with  $H_2SO_4$ , extracting with ether, shaking up the ethereal solution with soda, and again acidulating.—D. E. J.

*A Method for preparing "Helianthin."* (Orange III.) By R. Möhlau. Ber. Ber., 17, 1490.

THE sulphonic acid of dimethylamidoazobenzene, the helianthin (or Orange iii.) of commerce, have usually been prepared by means of the combination of paradiazo-benzene sulphonic acid, with dimethylaniline. (Ibid. 10, 528). When a solution of the hydrochlorides of aniline and dimethylaniline in equivalent quantity is treated with nitrous acid, the products of the resulting reaction are diazobenzene chloride and nitrosodimethylaniline hydrochloride; if, on the other hand, a nitrite be employed in place of the nitrous acid, and the solution made alkaline, the reaction takes a different direction, dimethylamidoazobenzene being formed according to the equation—



The product is easily sulphonated by dissolving it in 20 times its weight of concentrated sulphuric acid (30 %  $SO_3$ ) and allowing to stand 24 hours at the ordinary temperature. On pouring into water, neutralising with soda or ammonia, and allowing to cool, "helianthin" is obtained in well-formed orange-yellow plates. If in the above reaction the aniline be replaced by paratoluidine, the corresponding dimethylamidobenzeneazotoluene,  $Me_2 \cdot NC_6H_3 \cdot N : NC_6H_4 \cdot Me$ , is obtained. It crystallises from alcohol in golden yellow plates (m.p.  $168^\circ$ ). It may be distilled for the greater part without decomposition. By reducing agents it is resolved into paratoluidine and dimethylparaphenylenediamine. The sulphonated derivative could only be obtained by indirect means. It was prepared by the method of Neville and Winther (Ibid. 13, 1947)—viz., the action of the diazotated derivation of paratoluidine sulphonic acid ( $CH_3 : SO_3H : NH_2 = 1 : 3 : 4$ ) upon dimethylaniline. It is obtained in prisms of a dark violet colour. The sodium salt crystallises in orange yellow plates.—C. F. C.

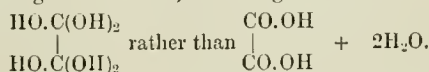
*On the Formation of Colouring Matters from Aromatic Acids.* Carl Zulkowsky. Monatshefte für Chemie, 5, 221.

IT was observed by Graebe and Caro that when aurin is heated with water to  $220-250^\circ$ , it is decomposed with formation of dioxibenzophenone and phenol; they attempted to prepare aurin from its decomposition product but without success (Berl. Ber. 1878, 1348). The author fused aurin with caustic potash, and obtained phenol and paroxybenzoic acid (Berl. Ber. 1877, 463), thus:  $C_{19}H_{14}O_3 + 4KOH = C_7H_4K_2O_3 + 2C_6H_5OK + 2H_2O$ . He was also unable to bring about the inverse reaction. When paroxybenzoic acid or salicylic acid is heated to  $140^\circ$  with phenol and sulphuric acid, the fused mass certainly becomes yellow, but it remains liquid even after heating for six hours, so that the reaction is very slow. The quantity of colouring matter is extremely small, but the fused mass gives the colour reaction of aurin with caustic potash. When, however, polyatomic phenols are heated with the above-mentioned acids, and probably with all aromatic acids, in presence of dehydrating agents, the reaction is very energetic. Previously coloured condensation products have been obtained only from aromatic aldehydes, chlorides, and phthalic anhydride; phthalic acid has indeed been employed, but it readily forms an anhydride. The investigations of the author have so far extended only to the action of benzoic and salicylic acids or resorcinol, orcinol, and pyrogallol, but the results point to the conclusion that other aromatic acids would yield similar products. The colouring matters have not been thoroughly investigated, but their general properties agree with those of the phthalines. *Benzoic acid and Resorcinol*.—A mixture of one molecule of benzoic acid to

two of resorcinol was placed in a flask with a quantity of granulated anhydrous zinc chloride equal to that of the resorcinol. The flask was provided with a perforated cork, through which passed a glass tube, to allow of the escape of gases and vapours. It was heated in a paraffin bath to 170-180° for 6 to 8 hours. The reaction commenced as soon as fusion took place, and the mass became at first yellow, then deep red. After cooling, the flask was filled with boiling water, and heated on the water bath until the fused mass became quite soft. The mixture was cooled, and the yellow liquid poured off. This treatment was repeated four or five times, when a residue resembling cantharides was obtained. The substance solidified when quite cold, and was crystallised by dissolving in a small quantity of boiling 50% alcohol, and allowing to cool. After standing some time, the crystals were filtered with the pump, and washed with 50 per cent. alcohol. The crystals are much less soluble in alcohol than the crude product; they form a gold yellow solution, with a green fluorescence, which is intensified by addition of ammonia or potash. According to theory the substance should be identical with Doebner's resorcinol-benzene, prepared from resorcinol and benzenyltrichloride. (Berl. Ber. 1880, 610.) A very good yield of the colouring matter was obtained by employing sulphuric acid as the dehydrating agent, the weight being equal to that of the resorcinol. The mixture was heated to 130-135° for 3 hours, and the residue treated with hot water as before. The chemically-combined sulphuric acid and other impurities were removed by boiling with dilute caustic soda in a porcelain basin, until a small residue only remained. The colouring matter was precipitated from the alkaline solution by addition of acetic acid, and was crystallised as before. *Benzoic Acid and Orcinol*.—A mixture of one molecule of benzoic acid to two molecules of anhydrous orcinol, with sulphuric acid in quantity equal to the orcinol, was heated for four hours to 130-135°. The mixture at once assumed a yellowish red colour. After treatment with water, the product was obtained as a steel-green brittle mass, with a metallic lustre. It was treated with caustic soda and acetic acid, and when thus purified its alcoholic solution possessed a beautiful gold-yellow colour, with a slight dark green fluorescence, which was intensified by addition of ammonia. It could be crystallised from glacial acetic acid, but no alcohol. *Benzoic Acid and Pyrogallol*.—A mixture of these substances with sulphuric acid in the usual proportions was heated to 120-130° for 2½ hours. The mixture became yellow, then brown, and lastly quite opaque. The colouring matter is soluble in hot water, and was purified by filtering its boiling aqueous solution, and allowing the filtrate to stand, when large crystals were obtained and were washed with cold water. The solution, filtration, and crystallisation were repeated, and the crystals dried *in vacuo*. They possessed a peculiar brown-red colour which was found to be due to a thin superficial layer, probably an oxidation product. The aqueous solution was brown-red, and became violet on addition of ammonia, but dirty when caustic potash was added. *Benzoic Anhydride and Resorcinol*.—The reaction was only brought about by addition of zinc chloride, but the yield was then as good as with benzoic acid. *Salicylic Acid and Resorcinol*.—Both zinc chloride and sulphuric acid were used as dehydrating agents and the temperature was maintained between 130 and 140°. In both cases the fused mass was rapidly coloured an intense red. At higher temperatures carbon-dioxide was evolved. With zinc chloride the reaction was complete in 6 to 8, with sulphuric acid in 4 to 5 hours. The substance, purified in the manner already described, was recrystallised from its solution in 50 per cent. alcohol, and formed dark red-brown crystals. The alcoholic solution has a reddish-yellow colour, with a green fluorescence, which becomes extremely intense on addition of ammonia. The colouring matter is probably the oxy-compounds of resorcinol benzein. The yield was smaller than in the previous cases. No experiments have yet been made with paroxybenzoic acid and polyatomic phenols, but it is probable that they will yield even better results, for the action of the para-compound on phenol is more energetic than that of salicylic acid.—S. Y.

*On a By-Product of the Manufacture of Aurin.* Part 11. By Adolf Staub and Watson Smith. Journ. Chem. Soc., xlv., July, 1884.

In a former paper by Alexandre Claparède and one of the authors (this Journal, August 29th, 1883, page 345), it was shown that the by-product referred to is formed by the direct union of 2 mols. of phenol with 1 of anhydrous oxalic acid. In a new experiment, by repeated recrystallisation from pure glacial acetic acid, and after drying in a vacuum over calcium chloride and quicklime, the crystals were obtained in a perfectly pure state. As already stated, this compound may be considered as the phenylortho-oxalic ether, which view is supported on the one hand by the instability of the substance, and on the other by the fact that the two naphthols form compounds analogous to it, though somewhat less readily. The experiment has also been tried with resorcinol, but up to the present time no success has attended the efforts to isolate any analogous compounds. Some hopes had been entertained of the possibility of obtaining either a compound in which 1 mol. of anhydrous oxalic acid unites with 2 of resorcinol, or one in which 1 mol. of oxalic acid is combined with 1 of resorcinol, both hydroxyl-groups of the resorcinol molecule in the latter case being engaged. All hope in this direction has not yet been abandoned, and should it be realised important evidence will thus be obtained of the so-called water of crystallisation of oxalic acid, existing in a form of union in the oxalic acid molecule, somewhat closer than water of crystallisation is usually believed to exist in—the evidence, in short, being in favour of crystallised oxalic acid being an ortho-acid, and being formulated—



The melting point of the phenyl ortho-oxalate, as determined in a capillary tube, was found to be 126 to 127°. It was altered by the presence of the smallest trace of moisture, and can be exactly determined only by exertion of the greatest care. By heating the substance in sealed tubes for five hours at about 180°, it was completely decomposed, yielding phenol, carbonic oxide, carbon dioxide, and water. On heating it with concentrated sulphuric acid for some time at 100-120°, aurin is obtained. To test the result of this reaction in presence of free phenol, 18 grams of the phenyl ortho-oxalate were mixed with 7 grams of phenol and 10 grams of pure concentrated sulphuric acid, and the mixture was heated for about 20 to 22 hours at 120-125°. The melt was poured into water, and the mixture boiled for some time to remove phenol and other soluble products. After boiling twice, crude aurin was left in a fairly pure state, and only 9 grams of the crude substance were thus obtained from 18 grams of the by-product. This small yield of aurin makes it probable that the presence of free phenol does not increase the formation of aurin. After standing for 24 hours, the aqueous solution was filtered off, and treated with barium carbonate in order to remove free sulphuric acid. By evaporation of the filtered solution, 23 grams of barium paraphenol-sulphonate crystallised out in small white needles. During the above-mentioned process of heating the by-product with phenol and sulphuric acid, the gases evolved during the reaction were caused to pass through water, and on testing this solution only traces of formic acid were detected. This result consequently does not bear out the theory involved in the equation expressing the formation of aurin in the former paper (*loc. cit.*), in which it appears that formic acid should be generated. To investigate this question more closely, a quantity of pure potassium phenolsulphonate (one of a series of fine preparations manufactured by Messrs. Charles Lowe and Co.) was mixed with the calculated proportions of anhydrous oxalic acid and concentrated sulphuric acid, and heated as in the usual way in the preparation of aurin. It was then found that if during any stage of the process the melt was allowed to cool, instead of the abundant sublimation of ortho-phenyl oxalate, only a few minute crystals of this compound were discernible, and this entirely out of proportion in amount to what is



found in the usual process of aurin preparation. The result thus obtained indicates that the ortho-phenyl oxalate plays no part as an intermediate product in the formation of aurin. The following experiment shows clearly how the substance may be formed during the process of the manufacture of aurin. Vapours of phenol were passed into a dry flask in which oxalic acid was being sublimed; an abundant deposit of the crystals of the orthophenyl oxalate was formed on the cooler portions of the interior of the flask. These results, then, coincide best with the theory expressed in Nencki's equation,  $3(C_6H_5.OH) + CH_2O_2 = C_{19}H_{15}O_3 + 2H_2O$  (see *J. pr. Chem.*, 25, 278), according to which formic acid plays the chief part in the formation of aurin. The endeavour was now made to solve the question, Does nascent carbon dioxide really act on phenol so as to yield aurin? as Zulkowski and others are inclined to think. (This Journal 111. [6], page 371.) To do this, the formation of the carbon dioxide was determined under the same conditions as in the aurin process. Two test-tubes were taken, and into one of them 2 grams of pure malonic acid and 5 grams of phenol were introduced and well mixed together, and then 10 grams of pure sulphuric acid; into the other the same quantity of malonic acid and sulphuric acid, without any addition of phenol. Both tubes with their contents were now heated at  $110^\circ$  to  $120^\circ$ , when the disengagement of carbonic acid began in both at about the same time and ended after about three hours. In both test-tubes, the whole contents had gone into solution in the sulphuric acid, which was thus merely coloured light brown. After saturation with soda, the two solutions showed as little indication, as regards any colouration produced, of the presence of aurin in the one tube as in the other—in short, not a trace of aurin was formed by the joint action of malonic and sulphuric acids on phenol. Of course in malonic acid  $(CO.OH).CH_2.(CO.OH)$ , under the circumstances named, the formation of carbonic acid is secured, to the exclusion of that of formic acid.

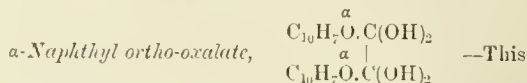
#### Compounds of Oxalic Acid with $\alpha$ - and $\beta$ -Naphthol.—

Experiments with the isomeric naphthols showed that these form compounds with oxalic acid similar to that which is formed by phenol.

The  $\beta$ -naphthol compound ( $\beta$ -naphthyl ortho-oxalate) ... ..  $\left\{ \begin{array}{l} \beta \\ C_{10}H_7O.C(OH)_2 \\ \beta \\ C_{10}H_7O.C(OH)_2 \end{array} \right.$

was obtained by mixing in the right proportions a warm glacial acetic acid solution of  $\beta$ -naphthol and oxalic acid, and heating the mixture with reversed condenser for some hours at the boiling point. It is best to employ a small excess of naphthol, which is subsequently removed with ease on washing the resulting compound with petroleum spirit. The presence of free oxalic acid in the product is thus entirely prevented. On cooling and standing for some time, the compound separates as a crystalline powder, which, after draining off the acetic acid, and drying over lime and calcium chloride, was boiled with petroleum spirit to remove free naphthol. The substance was then recrystallised from pure glacial acetic acid, and dried in the exsiccator as before. The final product is a perfectly white crystalline powder, melting with slight decomposition at  $167^\circ$ . (Perfectly anhydrous oxalic acid melts with decomposition, as we observed, between  $186^\circ$  and  $187^\circ$ , using capillary tubes in the melting point determination. The melting point of this acid is thus considerably higher than is generally stated in the text-books.) On distillation under the ordinary atmospheric pressure, it is, like the phenol compound, partially decomposed, and the presence of formic acid could be readily detected in the products of distillation by its pungent odour, and the mirror-like metallic reduction of silver from an ammoniacal silver solution. The residue in the retort consisted chiefly of  $\beta$ -naphthol. This compound is also formed, as well as the  $\alpha$ -naphthol compound, by heating oxalic acid and naphthol together at from  $120$ – $130^\circ$  for several hours. When however it is prepared in this way, it is difficult to separate the compound from free oxalic acid. So far

as the other properties are concerned, the substance behaves like the phenyl ortho-oxalate.



compound was prepared in a manner similar to that employed for the  $\beta$ -naphthol compound. It is, however, not so easily formed as the  $\beta$ -naphthol compound. The melting point of the substance is  $163^\circ$ , slight decomposition ensuing at that temperature. Its properties and appearance closely resemble those of the  $\beta$ -naphthol compound.—W. S.

#### Improvements in the Manufacture of Colouring Matters.

William Robert Lake. Communicated by Messrs. Léo Vignon and Co., Lyons, France. Eng. Pat. 5515, November 24th, 1883. Provisional protection only.

THE inventors claim the manufacture of the diazo-colours resulting from the action of diazotised amines, and their sulpho acids upon the various sulpho acids of alpha-naphthol. The amines mentioned are aniline, and its homologues, amidoazo-benzene, naphthylamine, &c. The authors give five methods for obtaining the alpha-naphthol sulphonic acids. (1) One part of naphthol and three parts monohydrated sulphuric acid are heated to a temperature not above  $25^\circ C$ . till the mixture is soluble in water. (2) The same mixture is heated for three hours at  $80^\circ C$ . (3) The same mixture is kept for three hours at  $120^\circ C$ . (4) Fuming sulphuric acid containing 20 per cent. of anhydride is used instead of the monohydrated acid in the foregoing mixture. (5) The alpha-naphthol-sulphonic acid produced by boiling the diazo-derivative of alpha-naphthylamine sulphonic acid with water. In the manufacture of these colours, which are stated to be yellow, orange, red, or violet, the solution of the diazo-salt is mixed with the alkaline solution of the naphthol-sulphonate in the usual way.—R. M.

#### Improvements in producing Brown Colours or Dyes upon Textile Fabrics or other Materials.

J. C. Newbarn. Communicated by "La Société P. Bonnet et Cie.," of St. Fons, Rhone, France. Eng. Pat. 5730, December 13th, 1883.

THE colours are produced in the fabric by first immersing in a warm bath containing a salt of a para-diamine (paradiamidobenzene preferred), and then oxidising with the usual 'aniline black' mixture, viz., potassium chlorate and a vanadium salt or bichromate. The shades produced vary from a cinnamon brown to a very dark brown.—R. M.

#### Improvements in the Manufacture and Application of Colouring Matters.

C. D. Abel. Communicated by the "Farbwerke vormals Meister, Lucius and Brüning," of Höchst on the Main, Germany. Eng. Pat. 5767, December 17th, 1883.

THIS invention claims the conversion of the azo-colours, whether soluble or insoluble in water, into soluble compounds by acting upon them with bisulphites. The colouring matter, in the form of a powder or paste, is dissolved or suspended in a suitable solvent, which may be water in the case of sulphonic acid salts, or alcohol in the case of colours insoluble in water, and the bisulphite added to the solution, which is kept warm till the conversion is complete. The compounds produced by the combination of the azo-colours with the bisulphites are termed "azo-S-colours," and the latter can be separated from the cold solutions either by crystallisation or by the addition of salt. The "azo-S-colours" are crystalline, and all dissolve in water with a yellowish red shade; by the action of heat, alkalis, or a nitrite they decompose into the original azo-colour. On the other hand they are not affected by the action in the cold of the neutral metallic salts used as mordants, nor by acetic or tartaric acids. The colours are applied by first impregnating the

fabric with the soluble dye in solution or in the form of paste, with or without a mordant, and development is then effected by means of heat, an alkali, or a nitrite, according to the particular fabric. Examples are given in the specification of the conversion into "azo-S-colours" of the following typical compounds:—(1) The azo-compound derived from alpha-naphthylamine and beta-naphthol. (2) The compound obtained from dichlorothiazophenol and ethyl-beta-naphthol. (3) The ordinary beta-naphthol orange obtained from diazosulphanilic acid and beta-naphthol.—R. M.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Apparatus for the Treatment of Wool and Mixed Fabrics, etc.* Eng. Pat. 5119. Oct. 29, 1883. Joshua Woodcock and Henry Webster.

THIS apparatus consists of an air-tight chamber, lined with lead or other suitable material, containing a series of travelling aprons and revolving rollers by which the wool or other material to be treated is kept constantly in motion. Hydrochloric acid gas is introduced for the purpose of destroying the vegetable matter contained in the wool. The chamber can be heated by a system of pipes and flues. Ammonia gas is sometimes introduced in order to neutralize the excess of acid remaining after the disintegrating process is complete. The apparatus can also be used for disinfecting purposes and for bleaching wool or other fibres. For bleaching wool sulphurous acid gas is introduced, and for jute or cotton chlorine gas. The specification is accompanied by drawings.—E. J. B.

*Carbonizing or Destroying Vegetable Matter contained in Woollen or Silk Fabrics.* Eng. Pat. 5223, Nov. 2, 1883. George and John Edwin Tolson.

IN this invention an apparatus is to be used consisting of a series of chambers through which the woven fabric is passed by means of rollers. The fabric is bleached in the first chamber and is then passed into the second chamber to be dried. From this it passes through four other chambers, where it is successively carbonized by means of hydrochloric acid gas, dried, neutralized and finally dried. The apparatus is fully described by means of drawings.—E. J. B.

*Waterproofing Fabrics.* By Ernest de Pass, 68, Fleet Street, London. Eng. Pat. 5242, November 5, 1883.

A BATH, heated to 90° C., is made of

Liquid Bordeaux turpentine	....	6 kilos. (13.20 lb.)
Tallow	.....	1.5 " (3.30 " )
Wax	.....	0.5 " (1.10 " )
Styrax (optional)	.....	0.2 " (0.44 " )

The articles to be rendered waterproof are immersed for a few minutes, then removed and passed through heated rollers to remove the excess of liquid.—J. M. H. M.

*Improvements in and relating to the Manufacture of Indiarubber and Guttapercha Coated Fabrics.* W. R. Lake, London. Communicated by N. S. White, Canton, Mass. U.S.A. Eng. Pat. 5802, April, 1, 1884.

THE improvements claimed are to be applied to the manufacture of indiarubber or guttapercha surfaced fabrics, and consist partly in providing the said fabrics with a permanent brilliant colouring, and in slowly curing the indiarubber or guttapercha, whereby the surface of the fabric remains soft, flexible, coherent, tenacious, and has a peculiar leathery feeling. Compositions for use in the said manufacture are also claimed. A filling compound, consisting of a mixture of sulphur, 20%, oxide of zinc, 50%, and suitable colouring material, 30%, is mixed with the dissolved indiarubber or guttapercha, to cure it and give it body and colour, and the surface of the fabrics is coated with this composition. It is essential in preparing this filling that the ingredients be thoroughly dried before mixing, and mixed dry. About five per cent. of

"aniline pigment" is always added to the filling mixture, this "aniline pigment" consisting of china clay, 50%, Westminister Paris white, 44%, precipitated silicate of soda, 5%; and aniline, 1%, is added to give brilliancy, tone and permanence to the other common pigment. When guttapercha is used, gum damar is added to prevent hardening of the surface of the cloth. The zinc oxide acts as a mordant for the colouring matter, and the filling, composed as above, provides for a slow and continuous curing of the indiarubber or guttapercha, thus giving the surface of the goods a soft leathery unctuous feeling. In some cases the first coating of the fabric may be made of indiarubber and gum balata, the succeeding ones consisting of guttapercha composition; the indiarubber remains elastic and flexible longer than the guttapercha, so that should the guttapercha coating crack, the india-rubber and balata layer prevents the crack extending through to the cloth and exposing its colour and texture.—W. M.

## VII.—ACIDS, ALKALIS, AND SALTS.

*On the Non-existence of Ammonium Hydroxide.* D. Tommasi. Compt. Rend. 98, 812.

THE author concludes from his law of thermal constancy (Berl. Ber. 15, 2355, 2732) that a hydroxide of ammonium does not exist in aqueous solution.—S. Y.

*On the Detection of Tartaric Acid in Citric Acid.* H. Athenstädt. Arch. Pharm. 22, 230-231.

THE author dissolves 0.5 gm. of the citric acid in 10 gm. distilled water, and adds 5 drops of this solution to 15 grms. lime water. The presence of tartaric acid is indicated by the turbidity of the liquid, which becomes more and more marked as the solutions mix. Shaking should be carefully avoided. The lime water must be so concentrated that 100 grms. will neutralize 4.5 cc. of normal hydrochloric acid solution, otherwise the calcium tartrate may not be precipitated.—S. Y.

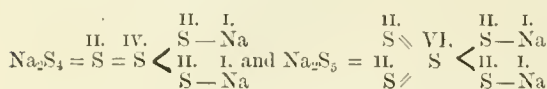
*On the Polysulphides of Sodium.* Dr. Heinrich Böttger, Annalen der Chemie, 223, 335.

BERZELIUS obtained the sulphur compounds of sodium and potassium by heating the carbonates with sulphur; and Schöne prepared those of potassium in the wet way. The author obtained the monosulphide by dissolving caustic soda in alcohol, filtering, dividing the solution into two equal portions, saturating the one completely with sulphuretted hydrogen and adding the other. The monosulphide was thus precipitated in small white crystals, which dissolved in the alcohol at 90° and separated by slow cooling in long prismatic crystals. The composition of the compound was found to be  $\text{Na}_2\text{S} + 5\text{H}_2\text{O}$ . The water was estimated by heating in a current of hydrogen; the sulphur by precipitation with copper sulphate, oxidation of the copper sulphide, and precipitation as barium sulphate. The sodium was estimated as sulphate. The polysulphides were prepared by first forming the monosulphide in the above manner, and then heating in presence of the alcohol with the requisite quantity of sulphur. The disulphide has the composition  $\text{Na}_2\text{S}_2 \cdot 5\text{H}_2\text{O}$ ; it is deposited from its alcoholic solution in sulphur-coloured crystals. The trisulphide was formed at 90°, and crystallised at -10° in dark gold-yellow crystals of the composition  $\text{Na}_2\text{S}_3 \cdot 3\text{H}_2\text{O}$ . The tetrasulphide was deposited from the alcoholic solution at -23°, or from a more concentrated solution at -15° in orange-red crystals, having the composition  $\text{Na}_2\text{S}_4 \cdot 8\text{H}_2\text{O}$ . When sodium monosulphide was heated with sulphur in quantity sufficient to form the pentasulphide, the tetrasulphide was deposited first, the pentasulphide being obtained from the mother-liquor at -5°, in dark orange-yellow crystals, which have the composition  $\text{Na}_2\text{S}_5 \cdot 8\text{H}_2\text{O}$ . The author found the same amount of water of crystallisation in the mono and tetra compounds as Schöne in the corresponding potassium compounds. Schöne obtained sodium tetrasulphide in a different manner with six molecules of water of crystallisation.—S. Y.



*On the Constitution of the Polysulphides of the Alkali Metals.* Dr. Heinrich Böttger. *Annalen der Chemie*, 223, 335.

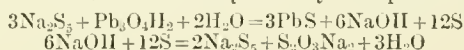
THREE views may be taken of the constitution of these compounds: (1) Sulphur may be considered as invariably divalent, and the alkali metal as monovalent, and formula of sodium pentasulphide, for instance, may be written  $\text{Na}-\text{S}-\text{S}-\text{S}-\text{S}-\text{S}-\text{Na}$ . (2) The valency of sulphur may be taken as constant, and that of the metal as variable, with all values from 1 to 5, or as monad, triad and pentad respectively. (3) The metal may be regarded as a monad, and the sulphur as dyad, tetrad or hexad, the tetrasulphide and pentasulphide being written like sulphurous or sulphuric acid, thus—



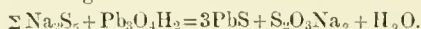
If this latter view be correct, sodium sulphite and sodium sulphate ought respectively to be formed when the tetra and pentasulphides of sodium are treated with lead hydroxide, thus—



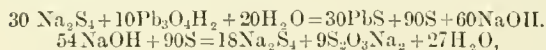
The author finds, however, that in both cases sodium thiosulphate is formed, while in that of the tetrasulphide the solution becomes alkaline from formation of caustic soda. The reactions are explained by the equations



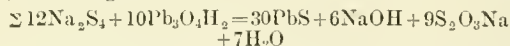
or, combining the two—



The sodium tetrasulphide behaves like pentasulphide, but the liquid remaining (because of the sodium hydrate formed) has an alkaline reaction. The reaction is exhibited as follows:—



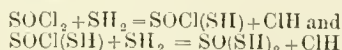
or, combining the two—



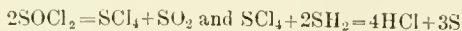
The author therefore concludes that sodium tetra and penta sulphide are not analogous to sodium sulphite and sulphate.—S. Y.

*Attempts to bring about the Combination of Sulphur with Sulphur.* Dr. Hugo Prinz. *Annalen der Chemie*, 223, 371.

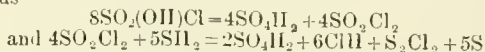
(1) It was thought possible that thionyl chloride and hydrogen sulphide might react on each other, according to the equations



but hydrochloric acid, sulphur dioxide, and free sulphur were formed, thus—

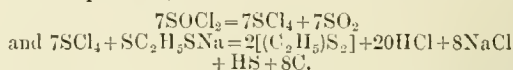


(2) By the action of hydrogen sulphide on sulphuryl hydroxy-chloride, sulphuric acid, hydrochloric acid, sulphur monochloride, and free sulphur were formed, thus—



(3) Sulphur was found to have no action on ethyl sulphate at 100°. (4) A mixture of ethyl sulphite and sulphur was heated to 200°, the tubes being opened from time to time to allow of the escape of sulphur dioxide. Besides this compound only ethyl ether and free sulphur were formed; and it was found that at 200° ethyl sulphite itself is decomposed with formation of ethyl ether and sulphur dioxide. (5) By heating ethyl sulphite with ethyl sulphide to 180°, the products formed were ethyl ether, sulphur dioxide, and unaltered ethyl sulphide. (6) The experiments of Böttger on the action of thionyl

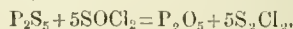
chloride on sulphur monochloride were repeated with concordant results, sulphur dioxide, hydrochloric acid, sulphur and carbon being formed. (7) Thionyl chloride and sodium mercaptide were found to yield ethyl disulphide and other products, thus—



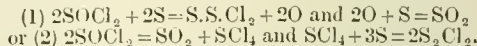
(8) By the action of ethyl sulphonic chloride,  $\text{C}_2\text{H}_5\text{SO}_2\text{Cl}$ , on sodium mercaptide at 150°, sulphur dioxide, hydrochloric acid, ethyl disulphide, and sodium chloride were formed, besides unaltered sodium mercaptide and free carbon. It is well known that diethyl sulphine oxide  $(\text{C}_2\text{H}_5)_2\text{SO}$  takes up oxygen with formation of diethyl sulphone  $(\text{C}_2\text{H}_5)_2\text{SO}_2$ ; but sulphur has no action on it even at 180°. It was thus not found possible to bring about the combination of sulphur with sulphur.—S. Y.

*On the Constitution of Sulphur Monochloride.* Dr. Hugo Prinz. *Annalen der Chemie*, 223, 355.

SULPHUR monochloride is looked upon by Carius (*Annalen* 106, 325, and 331) as sulphothionyl chloride, this view of its constitution being borne out by its supposed formation from phosphorus pentasulphide and thionyl chloride, thus—

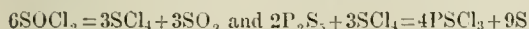


To test the truth of this view, the author attempted in various ways to obtain thionyl chloride by the replacement in sulphur monochloride of an atom of sulphur by oxygen, and conversely to prepare sulphur monochloride from thionyl chloride. Sulphur monochloride was heated with sulphur dioxide, but no reaction took place even at 100°. The action of antimony trioxide, arsenic trioxide, phosphorus trioxide and pentoxide, antimony pentoxide, arsenic pentoxide, selenium dioxide, boron trioxide, and silicon dioxide on sulphur monochloride was also investigated. The oxides of phosphorus, boron and silicon were unacted on, while in the other cases sulphur dioxide, the chloride of the element, united with oxygen, and generally free sulphur were formed. In no case was thionyl chloride produced; but there was always a complete interchange between the chlorine and oxygen. Hence, sulphur monochloride behaves rather as a mixture of sulphur tetrachloride and free sulphur ( $2\text{S}_2\text{Cl}_2 = \text{SCl}_4 + 3\text{S}$ ) than as sulphothionyl chloride. In order to study the action of thionyl chloride on sulphur and its compounds, 500 grms. of phosphorus pentachloride were treated with sulphur dioxide, and after repeated rectification 100 grms. of pure thionyl chloride, boiling at 76-78°, were thus obtained. It was found to be possible to convert thionyl chloride completely into sulphur monochloride by heating the former compound with sulphur, according to the equation  $2\text{SOCl}_2 + 3\text{S} = 2\text{S}_2\text{Cl}_2 + \text{SO}_2$ , in sealed tubes to 180°. The tube was opened from time to time to allow of the escape of sulphur dioxide, and after three days the conversion was found to be complete. The reaction may be looked upon as taking place in either of two ways;

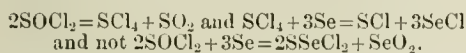


Böttger's researches on the action of thionyl chloride on ethyl sulphide agree with the latter view. The action of sulphur on other oxychlorides was studied with the following results: Sulphur does not act on phosphorus oxychloride even at 200°. When sulphur and acetyl chloride were heated to 190°, a few drops of a substance boiling between 120° and 130°, and smelling of garlic, were obtained; but almost the whole of the acetyl chloride remained unaltered. With benzoyl chloride there was no action even at 230°, nor was alcohol attacked at 200°. Acetyl anhydride behaved like acetyl chloride, a portion being carbonised, and a trace of a sulphur compound formed. It therefore appears impossible to substitute sulphur in the free state for oxygen in oxychlorides. An attempt was made to bring about the substitution by means of combined sulphur. Thionyl chloride and antimony trisulphide yielded, however.

antimony chloride, sulphur dioxide, and free sulphur. The action, described by Carius, of thionyl chloride on phosphorus pentasulphide was therefore investigated, and it was found that at 100° sulphur dioxide, free sulphur and phosphorus chloride were formed, but not a trace of sulpho-monochloride. At 150° a small quantity of this compound was formed by the action of the free sulphur on the thionyl chloride. Hence, thionyl chloride behaves like a mixture of sulphur dioxide and tetrachloride, and the reaction takes place thus :—



This view is also borne out by the action of selenium on thionyl chloride, when sulphur and selenium monochlorides and sulphur dioxide are formed, thus :—



Sulphur monochloride can, therefore, no longer be considered as sulphothionyl chloride.—S. Y.

*Grinding Caustic Soda.* Sydney Pitt. (A Communication from Francis Parkinson Harned, of Camden, N.J.) Eng. Pat. 4677, October 2, 1883.

To prevent the deliquescence of caustic soda during the operation of grinding at ordinary temperatures, the patentee adds to the broken mass from one to two per cent. of soda ash or carbonate of soda.—H. J.

*Kilns for Burning Limestone.* John Briggs, Buxton. Eng. Pat. 4876, October 13, 1883.

THE kiln is open at the top, and is made with a contraction or neck near the centre of its depth, and with a door at the lower end. On each side of the neck is an arched passage, provided with windows. These arched passages are entered by an archway from the outside. From these passages air pipes pass through the walls into the neck of the furnace to admit air, and between the air pipes are a series of retorts for the conversion of slack coal into coke, the gas driven off in the process passing into the furnace direct from the retorts. At the bottom of the neck are steam pipes for the admission of dry superheated steam. The limestone to be burnt is brought in tram-wagons and poured into the top of the kiln, no levelling, setting or packing being required; the limestone then descends without any fuel whatever being mixed with it, thereby producing good clean lime. The retorts are filled with coal or slack by the firemen working in the arched passages, after which the mouth of the retorts is tightly screwed up, and the heat of the material in the kiln drives off the gas out of the coal or slack, and the gas thus produced passes into the neck of the kiln, when, meeting with the air, combustion takes place. The air pipes act as sight-holes. Openings are also provided to admit fuel into the lower chamber of the kiln should the fire be allowed to go out at any time.—J. T.

*Improvements in the Manufacture of Chlorate of Soda.* Edmund Knowles Muspratt and George Eschellmann, Ph.D. Eng. Pat. 5183, October 31, 1883.

THE present invention relates to the production of chlorate of soda, and consists in the employment of magnesia in conjunction with chlorate (in a manner similar to that now adopted in the production of chlorate of lime), so as to obtain chlorate of magnesia and chloride of magnesium. The products thus resulting are then treated with either caustic soda, or with carbonate of soda, or with mixtures of the same, so as to

produce corresponding compounds of soda, viz., chlorate of soda and chloride of sodium, and also either magnesia or carbonate of magnesia or mixtures of the same.—H. J.

*Improvements in the Manufacture of Chlorate of Potash, and in the Treatment of the Mother-Liquor resulting therefrom.* Edmund Knowles Muspratt and George Eschellmann, Ph.D. Eng. Pat. 5163, Oct. 31, 1883.

MAGNESIA is suspended in water and treated with chlorine gas. The produce is liquor containing magnesium chlorate and magnesium chloride in the proportion of about five to five and a half equivalents of the latter to one of the former. This liquor is then concentrated to from 75° to 80° Twaddell, and magnesium chloride crystallises out on cooling, leaving a liquor containing one equivalent of magnesium chlorate to about four equivalents of chloride. This liquor is then heated with potassium chloride, and the potassium chlorate formed is crystallised out. From five per cent. to ten per cent. potassium chlorate remains in solution, and its chlorine is liberated by the addition of hydrochloric acid to the mother-liquor. This mother-liquor is then neutralised with magnesium carbonate, and may be concentrated for sale, or converted into magnesia, and used over again.—H. J.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Improvements in the Manufacture of Cements, etc.* E. Robbins, London. Eng. Pat. 4773, Oct. 8, 1883.

THE patentee lays claim to thirty inventions or improvements in the manufacture of cements and combination of materials for architectural and like purposes, these inventions to be employed as a means "to carry out a complete change in coloured dressings and other work." A concrete cement of the kind most in demand is composed of various limestones, cobble chalk, cobble gypsum, or carbonaceous and argillaceous sulphates of lime. These materials may be used with such binding agents as ordinary cements, Portland, Parian, or Keen's cements, or with alkaline aluminates or silicates. They are "rendered durable by immersion in strong solutions of copperas and other chemicals, or by other treatment." Ordinary colours or chemical dyes may be incorporated with this improved concrete. The patent also refers to processes for rendering buildings weatherproof and fireproof, this induration being effected by the action of alkaline silicates on the building material. Some of the claims and instructions are rather vague; as, for example, "in preparing the fibrous materials for spinning, weaving, or otherwise working woven textile fabrics, paper, or other like materials, I sometimes charge or compound fibrous or other neutral ingredients with about 2½ per cent. more or less of an active agent, such as quicklime, or other chemicals, to which may be added an equal percentage of pure silicate soda or potash, or silicate held in suspension, with fired carbonaceous or argillaceous sulphate of lime, which is the most ready and convenient for use, the active and effective condition of the solubility of the silicate, as required for the same and other purposes of a similar kind." With regard to this vagueness, the patentee remarks that in "the absence of sufficient knowledge for the application of concrete to building purposes, coupled with the imperfect knowledge in this branch of industry, it is difficult to give any precise particulars or proportions of the materials employed in carrying out my improvements, with few exceptions."—A. R. D.



## X.—METALLURGY, MINING, Etc.

On the Gases evolved from Steel Castings. Dingl. Polyt.

Journ. 251, 83, 1884.

IN opposition to Pourcel, who ascribes the separation of gas in steel castings to carbonic oxide, Müller (*Stahl und Eisen*, 1882, 532) adduces his analyses of gases obtained by boring steel blocks under water. He found that these gases almost invariably consisted of nothing but nitrogen and hydrogen. Pourcel holds that iron which contains carbon has the power of dissolving hydrogen in proportion to the amount of manganese present; on the other hand, Troost and Hautefeuille shewed in 1874 that so long as a steel contained traces of manganese the solubility of hydrogen therein was determined by the amount of silicon present. On the ground of a large number of analyses, Pourcel likewise holds that silicon, as well as manganese, plays an important part in the deoxidation of the Bessemer—or Martin—bath by spiegel iron, ferro-manganese, or silicite. He has found that from one-third to one-half of the silicon, added in the form of silicite, passes into the slag, and therefore, in opposition to Müller, who maintains that only carbon and manganese are consumed in the deoxidation, holds that silicon and manganese are simultaneously absorbed. That silicon does possess a deoxidising power is further shown by Richards, of Eston, in connection with the Thomas-process. He added silicite to an overblown bath, and no separation of gas took place. Subsequent addition of rich spiegel iron caused no evolution of carbonic oxide, hence he considers that by a previous addition of silicite, evolution of gas, when the ferro-manganese is thrown in, may be completely avoided. The presence of silicon in steel, made by the German-Bessemer process, is ascribed by Müller to alterations in the affinity of carbon and silicon for oxygen, according to the temperature; by Pourcel, however, to a reduction of silicic acid in the lining by the carbon contained in the iron and manganese. In a direct experiment, conducted by over-heating a steel bath, free from silicon, on the Martin-hearth, with acid lining, he found that 0.003 to 0.004 per cent. of silicon was dissolved in a few hours. The porosity sometimes observed in blocks of German-Bessemer steel, even when silicon is present, is to be ascribed to the incomplete mixture of the spiegel iron with the bath, so that the oxide of iron is not reduced in the converter, but in the moulds. In such cases the steel is blown, and more or less "red-short." This observation is confirmed by Helmholtz. In the Martin process nothing of this kind occurs. In order to obtain conclusive evidence as to the reduction of oxide of iron and carbonic oxide by silicon, an acid Bessemer bath was overblown to such a point that an addition of spiegel iron would cause a dangerous reaction and destruction of the crucible. Instead, then, of adding spiegel iron, a raw iron, with 8 per cent. of silicon, about 2.5 per cent. of carbon, and little or no manganese, was applied in proportion to the reducing power of silicon, as compared with that of manganese. The reaction proceeded quite quietly, no evolution of gas took place, and the steel was free from pores, but red-short, owing, as analysis showed, to an intramolecular dispersion of silicite of iron through the steel. If, in place of silicon iron, a silicon-manganese iron be applied, no dangerous reaction will occur in the converter, and the steel will be free from blow-holes and ductile. Troost and Hautefeuille have found that when cold, silicon iron has the power of occluding a larger proportion of carbonic oxide than manganese iron. Müller has examined several samples of basic steel by boring blocks under water with a chamfered tool, so that shavings were obtained which would allow of all gas from the actual pores to be collected together, but little from the intramolecular spaces of the solid metal. The following table summarises his results:—

Description of Steel.	Percent- age of Gas.	Composition of Gas.		
		CO.	II	N.
I.				
Soft (Thomas') steel, prepared with 2.5 per cent. of ferro-manganese of 35 percent. Castings spirted considerably, and the blocks contained many radial pores .....	36	0.6	85.4	11.3
II.				
Basic metal finished without any addition. Quiet in pot and mould, and spirted but little. The blocks contained few radial pores .....	20	0	61.5	35.4
III.				
Thomas' steel, prepared with 5% of silicite of 14% and 2.5% of ferro-manganese of 70% per cent. The steel rose quietly. The blocks were moderately porous .....	22	0.4	86.4	12.7
IV.				
Thomas' steel, prepared with 5% of silicite of 14%. The steel rose quietly, and the blocks contained very few pores .....	6	0	54.7	45.3

It is worthy of remark that in spite of the large amount of silicon and manganese added, the steel only contained traces of silicon and 0.4 per cent. of manganese. It will also be observed that with a decrease in the total quantity of gas contained, there is an increase in the percentage amount of nitrogen therein. For the examination of the gases evolved during the solidification of raw iron, samples of this were re-melted in a cupola furnace and cast in small moulds. (I) Grey Bessemer raw iron evolved much gas, the block however was solid and structure granular. (II) Spiegel iron evolved much gas, the block was solid and the structure crystalline. (III) Thomas' raw iron with 3 per cent. of phosphorus evolved less gas, the block was free from blow holes. The analyses of the iron and gases collected are as under:—

	IRON.		
	I.	II.	III.
Carbon .....	3.638	4.18	3.099
Silicon .....	1.63	0.253	0.203
Manganese .....	1.93	7.37	0.736
GAS.			
Carbonic oxide....	36.1	48.1	37.1
Carbonic acid ....	3.7	1.2	3.3
Hydrogen .....	53.3	48.9	43.7
Nitrogen .....	3.6	2.0	14.2
Oxygen .....	0.3	0.1	1.2

In Bochum, raw iron with a certain amount of silicon and manganese is melted in the cupola furnace. The carbon at once commences to burn. One half of the silicon oxidises at the commencement, the remainder appears to remain unconsumed until nearly the end of the process, when, the carbon being eliminated, it burns energetically together with the manganese. The blown bath is then deoxidized and carburated by the addition of melted spiegel iron which causes a vigorous evolution of carbonic oxide. On emptying the metal into the collecting pot inflammable gases are again given off. In casting, the metal is very quiet and solidifies to blocks absolutely free from blow-holes. It is worthy of remark that more

gas is evolved from spring than from rail steel. The analytical results are as under:—

Metal	Rail steel		Spring steel	
Carbon .....	0.23	0.23	0.18	0.53
Silicon .....	0.15	0.25	0.15	0.25
Manganese .....	0.50	0.60	0.80	0.40
Phosphorus .....	0.06	0.08	0.06	0.08
Gases				
Carbonic oxide .....	35.5		21.1	41.7
Carbonic acid .....	7.0		5.8	0.4
Nitrogen .....	11.3		29.5	37.9
Hydrogen .....	45.3		35.1	19.1
Oxygen .....	1.0		5.6	0.7

There are also some points worthy of remark in connection with the gaseous contents of Bessemer metal which has been completely decarburized and contains a certain amount of oxide of iron. In the absence of carbon, the metal cannot saturate itself with carbonic oxide on account of oxidation, neither, in the presence of an excess of oxygen, can there be any formation of carbonic oxide by reduction of atmospheric carbonic acid. As the following analysis shows, the separated gases consist mainly of nitrogen and hydrogen:

Carbonic oxide .....	15.6	17.5
Hydrogen .....	43.4	51.1
Nitrogen .....	33.7	29.1
Carbonic acid .....	1.8	2.2
Oxygen .....	5.2	0.8

The metal rose in the moulds, evolved gas briskly, and the blocks contained a porous zone at the surface and separate blow-holes in the mass. Completely decarburized, red-short Martin metal was found to contain a gaseous mixture richer in carbonic oxide than the above, but poorer in nitrogen, facts probably owing to the layer of slag at the surface preventing any considerable absorption of nitrogen and hydrogen from the furnace gases and favouring the steady and gradual combustion of the carbon and saturation of the metal with the carbonic oxide so produced. Gases obtained from Martin metal were found to possess the following composition:—

Carbonic oxide .....	36.5	39.0
Hydrogen .....	26.7	36.9
Nitrogen .....	30.5	17.8
Carbonic acid .....	1.3	2.9
Oxygen .....	4.7	3.0

In the case of basic steel, the reaction in the converter on the addition of spiegel iron is as a rule by no means violent, but a considerable evolution of inflammable gases takes place first on pouring the metal into the collecting pot and again on filling the moulds. In spite of this, the formation of pores is seldom serious, and the blocks are usually compact and free from blisters. The following analyses were made upon samples of the gases separated from a soft basic steel, in which the deoxidation and carburization had been effected by the addition of 2% of ferromanganese of 95%:—

Carbonic oxide .....	50.7	55.8	75.1	57.7	47.0	75.4
Hydrogen .....	2.0	4.6	6.0	4.8	5.5	6.2
Nitrogen .....	44.7	39.3	19.0	30.5	46.0	17.6
Carbonic acid .....	—	—	—	—	0.1	0.3
Oxygen .....	2.6	0.6	0.5	—	1.3	0.5
	100.0	100.3	100.6	100.0	100.2	100.0

The examination of the gases evolved from basic rail steel during the process of casting and of those separated in the solid metal shows that, in the first case, carbonic oxide is the main constituent, but that, in the second case, the composition of the gases more nearly approaches to that of the gases separated in acid steel:

Basic Rail Steel.	Composition of Gas—	
	During Casting.	After Solidification.
Carbonic oxide .....	81.7	54.1
Hydrogen .....	8.2	38.6
Nitrogen .....	9.5	3.5
Carbonic acid .....	0.6	3.8
	100.0	100.0

It is thus evident that the brisk evolution of gas from basic steel is mainly due to carbonic oxide, whilst with raw iron, Bessemer and Martin steel, carbonic oxide is usually accompanied by large amounts of hydrogen. The evolution of carbonic oxide by metal in the moulds, Müller does not ascribe to deoxidation, for he considers

that although it is possible that when cold ferro-manganese is added to the contents of the converter the reaction may not be complete at that stage, yet the deoxidation is certainly complete when the metal reaches the collecting pot. He rather considers that the steel becomes supersaturated with carbonic oxide at high temperatures and evolves the same on cooling in the moulds. This view of the actual solution of carbonic oxide appears to be supported by the observation that even very small drops of molten metal will, on cooling, suffer considerable disturbance, will spirt and be dispersed in fragments. The main results of Müller's experiments may be summarized as under. The "rising" of steel is to be ascribed to a separation of hydrogen, but "spitting" is due to an evolution of carbonic oxide which the metal is unable to keep dissolved at its point of solidification. Basic metal without any addition contains hydrogen and nitrogen with but little carbonic oxide, rises but does not spit. By addition of spiegel iron or ferromanganese, formation of carbonic oxide takes place, the steel becomes saturated with this gas, which is given off on the cooling of the casting. By the addition, however, of silicite, the formation of steel is effected without disengagement of carbonic oxide; the steel in this case rises and evolves hydrogen. Brüstlein (*Annales Industrielles*, 1883, 551) takes a different view to either that of Pourcel or Müller as to the function of silicon in preventing the formation of pores. He considers that both silicon and manganese render a steel more pervious to occluded gases, which can thus escape without giving rise to blisters or spitting. The separation of hydrogen which takes place on cooling steel castings, Brüstlein explains by assuming that, at about the melting point of steel, iron and steel can form with nascent hydrogen a compound which at a red heat suffers dissociation.—W. D. B.

*Manufacture of Paving Blocks from Furnace Slag.* C. J. Dobbs, Middlesbro'. Eng. Pat. 3870. August 9, 1883.

THE inventor claims the method of annealing the blocks exclusively by means of their own initial heat, by enclosing them immediately after they have solidified in kilns or chambers of moderate dimensions that are only heated by a heat radiated from the blocks. The use of a series of kilns or chambers arranged side by side and communicating with each other, so that the heat of the slag blocks contained in one chamber may be conveyed into the contiguous chamber or chambers, so as to heat it or them preparatory to receiving a charge of slag-blocks.—A. H. A.

*Treatment of Ores, &c., containing Antimony, by the Wet Process.* James Beveridge, Runcorn. Eng. Pat. 4758. October 6, 1883.

THE process is applicable to ores and substances containing compounds of antimony which are soluble in hydrochloric acid. Ores containing 20 per cent., more or less, of antimony in the form of sulphide ( $Sb_2S_3$ ) are preferred. To every cubic foot of hydrochloric acid of sp. gr. 1.14 is added a quantity of ore representing five pounds weight of metallic antimony. The finely ground ore and acid are heated to the boiling point in any convenient way. Less than 1 per cent. of antimony is left in the gangue after washing. The solution of antimony trichloride is treated with 4-5½ times its volume of water, which precipitates about 95 per cent. of the antimony as oxychloride; on heating to 115°-155° F. the oxychloride quickly settles out. The waste liquor from this operation is treated preferably in scrubbing towers with part of the sulphuretted hydrogen evolved during the first operation, the antimony contained in the weak liquor is thus obtained as sulphide. From the weak liquor the excess of sulphuretted hydrogen is removed by passing air through it, and then adding a little chlorine or other oxidising agent. The resulting weak acid is then used in the condensing towers of alkali works instead of water. The oxychloride produced is reduced to metal by fusing with common salt, sodium carbonate, and coal; an exceptionally pure metal is the result.—J. T.



*Metallurgical Furnaces.* John Thomson King, Liverpool. Communicated by Simeon Bissell, Pittsburgh, U.S.A., Eng. Pat. 4779. October 9, 1883.

A REVERBERATORY furnace with saucer-shaped hearth is proposed; the usual fireplace is preferably replaced by a gas generator. An iron barrel filled with loosely stacked refractory brickwork, and divided longitudinally into two or more compartments, is inserted between the hearth and the chimney; this serves as regenerator. The products of combustion pass from the hearth through the upper compartment to the chimney, whilst air passes through the lower compartment, thence underneath the hearth to the furnace bridge, where it meets the gases from the generator. A partial revolution of the barrel charges the chambers without affecting the direction of the gas or air current. Dipping tuyers are also proposed by which a current of air can be directed on to the surface of the bath in the hearth, in such a manner as to produce a swirling motion in the contents of the hearth. It is claimed that pig iron can be rapidly converted into fibrous iron or into mild steel by the use of this apparatus.—J. T.

*Obtaining Aluminium from its Ores, &c.* Henry Arthur Gadsden, London; communicated by Emerson Foote, New York, U.S.A. Eng. Pat. 4930. October 16, 1883.

SODIUM vapour and a volatile compound of aluminium are generated simultaneously in two separate vessels, and the vapours thus obtained are mingled in a third vessel, wherein they react upon each other, producing metallic aluminium, which is upon the bottom of the vessel. Suitable aluminous material is calcined, ground, and mixed with about half its weight of sodium chloride and half its weight of carbonaceous matter. This is made into balls with water, dried, and heated to redness in a retort charged with chlorine; a double chloride of aluminium and sodium is produced. This double chloride passes into a suitable receiver, into which, at the same time, sodium vapour is also entering, the latter being produced by heating a mixture of sodium carbonate with charcoal. The given aluminous material, might be converted into any other compound of aluminium capable of being vapourised, as for example, the double chloride of aluminium and potassium, the simple chloride of aluminium, the fluoride of aluminium, or the double fluoride of aluminium and sodium or potassium, &c. It is also evident that potassium vapour could be employed in place of sodium vapour.—J. T.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

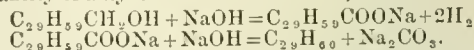
*On the Chemical Composition of Carnauba Wax.* H. Stürcke. *Annalen der Chemie*, 223, 283.

CARNAUBA wax is obtained from Brazil, and is a substance of considerable commercial importance, but its chemical composition has been incompletely investigated, and the results obtained by different observers are somewhat contradictory. Bérard (*Bull. Soc. Chim.* [1] 9.41), Story Maskelyne (*Chem. Soc. Journ.* 7, 87), and Pieverling (*Annalen*, 183, 344), agree in stating that the wax consists chiefly of the ether of an acid,  $C_{27}H_{54}O_2$ , with myricyl alcohol,  $C_{36}H_{72}O$ ; but Bérard finds a considerable amount of free cerotic acid,  $C_{27}H_{54}O_2$ , present, while Story Maskelyne believes that about 30 per cent. of the wax consists of free myricyl alcohol. Pieverling, from his analytical results, doubts the presence of free alcohol. Two samples of wax were examined by the author, one obtained from an English druggist, the other from Messrs. Gehe and Co., in Dresden. Their properties were precisely similar, and they were therefore not investigated separately. The melting point was 83–83.5°. Some wax was digested with boiling alcohol, in which it is partially soluble, and equal weights of the dissolved portion, of the undissolved, and of the original wax, after

saponification with caustic soda, were extracted with petroleum boiling between 75° and 90°. The quantities of extract were:—Original wax, 61; dissolved portion, 78.4; and insoluble portion, 54.1 per cent. Both the free acid and the alcohol are more soluble in alcohol than the ether, and hence if free alcohol be present, the extract from the portion soluble in alcohol should be larger than the others, which was found to be the case. The ether,  $C_{27}H_{54}O_2 \cdot C_{36}H_{72}$ , would give, after saponification, 52.77 per cent. of extract, hence the undissolved portion consists mostly of this ether. Free alcohol, and not acid, is therefore present in the wax. The free alcohol and hydrocarbons were separated from the ethers in 1,200 grms. of the wax by boiling it with  $5\frac{1}{2}$  litres of alcohol, pouring off the hot solution and allowing it to stand. 350 grms. of substance, mostly melissyl alcohol and ether, separated on cooling. The portion soluble in alcohol was saponified with 25 grms. caustic soda, and the mixed soap and alcohols carefully washed and dried. The mass was granulated and extracted in fractions with petroleum—boiling, for the first fractions at 75–90°, for the later ones at 110°. The 850 grms. of wax left undissolved by the alcohol were similarly saponified and extracted, and the alcohols were found to have the same melting points ( $82^\circ$  to  $87^\circ$ ), except a very small portion melting as high as  $95^\circ$ . A third portion of wax was directly saponified, dried, and extracted in 20 fractions, the melting points rising from  $81.5^\circ$  to  $99^\circ$ . Altogether 2,800 grms. of wax yielded 1,550 grms. of extract. The alcohols were then subjected to a very extended series of fractionations in the following manner:—The portions 1–4 were warmed with petroleum until partly dissolved, and pressed while hot through linen. The residue was added to the fractions 5–12, and these treated in the same manner. Three main fractions were finally obtained: (1) melting at  $77$ – $79^\circ$ , but still impure, amounting to 1.5 to 2 per cent. of the wax employed; (2) melting at  $86$ – $86.5^\circ$ , and exceeding 45 per cent. of the wax; (3) a small quantity (1 to 1.5 per cent.) melting at  $100$ – $101^\circ$ .

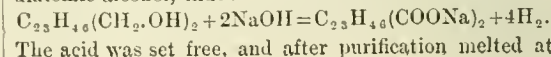
*Fraction 1.*—By repeated digestion with alcohol a hydrocarbon was left behind melting at  $59$ – $59.5^\circ$ , and containing 84.98 per cent. of carbon, and 15.18 of hydrogen. The portion soluble in alcohol was deposited in fine crystals, which, after purification, melted at  $76^\circ$ , and possessed the composition  $C_{26}H_{54}O$  or  $C_{27}H_{56}O$ . Only about 1.5 grm. was obtained. Of this 1.1 grm. was heated with soda lime and the acid separated. The acid was precipitated from its alcoholic solution in two fractions with magnesium acetate, and each portion treated with hydrochloric acid. The melting point of each portion of acid so obtained was practically the same,  $75.4^\circ$  and  $78.1^\circ$ , hence the substance was homogeneous. The formation of the acid is represented thus:  $C_{26}H_{53}CH_2OH + NaOH = C_{26}H_{53}COONa + 2H_2$ .

*Fraction 2.*—After purification this portion crystallised from petroleum in lustrous white leafy crystals, melting at  $85.5$ – $85.7^\circ$ , and solidifying at  $85.3^\circ$ . The substance possessed weak electrical properties. After fusion it solidified in concentric wavy rings. It was found on analysis to be pure myricyl alcohol. A portion was heated with soda-lime, when melissic acid and a small quantity of a hydrocarbon were formed, thus:



The acid was set free, and after purification was analysed; it melted at  $89.9$ – $90.2^\circ$ . The formula  $C_{30}H_{60}O_2$  was found. The lead and silver salts were also prepared and analysed.

*Fraction 3.*—After repeated recrystallisations the substance was obtained as a pure white crystalline powder, less electric than myricyl alcohol, and melting at  $103.5$ – $103.8^\circ$  ( $105^\circ$ , Story Maskelyne). The analytical results agreed best with the formula  $C_{25}H_{50}O_2$ , but Story Maskelyne proposes the formula  $C_{30}H_{58}O_3$ . The substance was heated with soda-lime, and the hydrogen collected. One molecule of the substance gave 4 molecules of hydrogen, hence the substance must be a diatomic alcohol, thus:



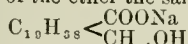
102.5°. The formula of the lead salt was found on analysis to be  $C_{23}H_{46}(CO)_2Pb$ .

*The Acids of Carnauba Wax.*—The different portions were united and boiled with alcohol. The greater part dissolved, and the solution was filtered and allowed to cool. The purified sodium salts were treated with hydrochloric acid, and melted with pure water. The mixture was extracted in 12 fractions with petroleum, the melting points rising from 72° to 86°. The residue was mixed with that from the alcohol, decomposed with hydrochloric acid, and fractionated as before. The final residue was now very minute. Fractionation of the acids by precipitation with magnesium acetate gave very abnormal results, and hence a combination of this method with that of partial solution of the free acids in petroleum was adopted—three fractions were finally obtained (α) (1) Melting point 70°, and (2) melting point 72–74°; (β) melting point 76–79°; (γ) melting point 100°. The second fraction was the largest, the first was much smaller, while the third amounted only to 8–10 grms.

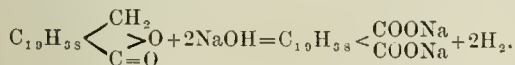
Fractions α and β were subjected to a series of fractional precipitations with magnesium acetate until acids were obtained with constant melting points. The acid from α melted at 72.5°, and that from β at 78.8°. From the fraction γ a small quantity of an acid melting at 82–84° was obtained; the residue melted at 103–104°, and solidified at 82–84°. The substances from fractions α and β were well defined acids of high molecular weight; that from γ had no very characteristic properties. The two acids resembled fatty acids, but they could not be crystallised, although Story Maskelyne states that he obtained them in the form of crystals. The analysis of the acid melting at 72.5° and of its lead salt gave the formula  $C_{24}H_{48}O_2$ ; the copper, zinc, and magnesium salts were also prepared. The acid melting at 78.8–79°, which is the chief acid component of carnauba wax, was found by analyses of the acid and of its lead salt to have the composition  $C_{27}H_{54}O_2$ , and it is either identical or isomeric with cerotic acid. The copper, silver, nickel, and magnesium salts of the acid were prepared. When the alcoholic solution of the substance melting at 103–104°, and obtained from fraction γ, was treated with magnesium, copper, or lead acetate, no salt was precipitated. By boiling with alcoholic potash, however, the potassium salt was obtained. After purification the acid was again liberated, and melted at 103.5°. The composition of the compound was found to be  $C_{21}H_{40}O_2$ . It was heated with soda-lime, when two molecules of hydrogen were evolved, which would be the case if two aldehyde or one alcohol group were present, but it is more probable that the substance is a lactone, containing the group— $C=O$



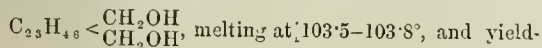
By saponification of the ether the salt



would be formed, and by the decomposition of the sodium salt the lactone and not the acid would be liberated, and the magnesium salt would not be precipitated by treatment with magnesium acetate. By heating with soda-lime a dibasic acid was formed, thus:



It melted at 89.5–90°, and its composition was proved by the analysis of its lead salt. The following substances have thus been isolated by the author:—(1) a hydrocarbon melting at 59–59.5°; (2) an alcohol,  $C_{26}H_{54}CH_2OH$ , melting at 76°; (3) myricyl alcohol,  $C_{29}H_{58}CH_2OH$ , melting point 83.5°, yielding melissic acid,  $C_{29}H_{58}COOH$ , melting point 90°; (4) a diatomic alcohol



ing the acid  $C_{23}H_{46}(CO_2H)_2$  with the melting point 102.5°; (5) an acid  $C_{23}H_{47}COOH$  melting at 72.5°, and isomeric with lignoceric acid; (6) an acid,  $C_{26}H_{54}COOH$ ,

melting point 79, identical or isomeric with cerotic acid;

(7) the γ oxy acid  $C_{10}H_{18} < \begin{array}{c} CH_2OH \\ COOH \end{array}$ , yielding the lactone  $C_{10}H_{18} < \begin{array}{c} CH_2 \\ >O \end{array}$  melting at 103.5°, and by heating with soda-lime the dibasic acid  $C_{10}H_{18}(COOH)_2$  melting at 90°.—S. Y.

*Processes for Solidifying liquid or semi-liquid Fatty Acids, etc.* E. A. Brydges, Berlin. (A communication from A. Marix of Paris.) Eng. Pat. 4040, August 21, 1883.

THE inventor aims at the solidification and transformation of mineral, vegetable and animal oils into solid fatty acids. This is effected by "heating the fatty acids in a suitable vessel by means of free steam with continuous agitation of the mass by means of air introduced through a system of injector-nozzles or in other appropriate manner; successively adding about 8 to 10% common or 5% concentrated nitric acid during the agitation of the mass; maintaining a temperature of about 100° for the space of about one hour, a continuous agitation of the material taking place; successively adding about 1% of starch or othersimilar product and continuing the aforementioned agitation; continuing the agitation for about 2 hours, raising the temperature to about 120°, and then reducing the same to about 70°; washing the product so obtained until all trace of acid is eliminated; treating the material in a suitable apparatus with a like quantity in weight of water strongly saturated with lime, caustic potash, ammoniac or other equivalent material; heating the product to 300° and maintaining this heat for about four hours, or employing a patented automatic circulating apparatus and working with a continuous vacuum, so that the heat employed does not exceed 160°; washing the product so obtained; then placing the same in a suitable apparatus and allowing the water and chemical products to settle or subside out of the same; treating this product with about 1% of sulphuric acid, and energetically agitating the same; distilling the product in a patented distilling apparatus by employing a vacuum; thus obtaining a pure white mass with very high melting point."—A. H. A.

*Separation of Glycerine from Fatty Substances.* C. Rumble and F. Sear, Battersea. Eng. Pat. 4264, Sept. 5, 1883.

THE fatty substances are mixed with 5 to 8% of a zinc or magnesium soap, and about one-third of their weight of water. The mixture is then subjected to the action of high-pressure steam, at a temperature of about 368° F. The glycerine set free can be separated from the "sweet water" in the ordinary way, while the soaps formed may be decomposed by a mineral acid, and the zinc or magnesium salt reproduced from the aqueous liquid by addition of a potassium or sodium soap.—A. H. A.

*Manufacture of Glycerine.* John Imray, London. Communication from T. A. F. Bang, Paris. Eng. Pat. 4593, Sept. 26, 1883.

THE inventor avoids the introduction of many of the impurities of glycerine by employing for the saponification lime, which has been previously well washed, to free it from chlorides and other soluble salts; similarly the limestone used for neutralising the product of the acidification process should be selected quite free from chlorides; and all the water employed in the process should be distilled. In operating by saponification, the glycerine liquor is separated from the decomposed fat, and cooled, when the greater part of the fatty acid separates as an emulsion. The clear liquor is then evaporated with addition of a small quantity of white stearic acid, which is said to have the effect of decomposing the calcium salts of the volatile fatty acids, which acids escape with the vapour. Small quantities of stearic acid are added whenever the scum tends to solidify. The calcium stearate formed has the effect of separating the various impurities.



The liquor is evaporated till it has a density of 22-24° Baumé, at 60° F., and then allowed to cool slowly, when the glycerine is found very pure and clear, and of good flavour, under the crust of stearic acid and stearate of calcium. It may then be further concentrated to 25-30° B., or when intended for making dynamite it is treated with a very small percentage of pure animal charcoal, concentrated to 30° B., and filtered. When the glycerine is required very pure and white for food purposes, a little more charcoal should be added, and the traces of lime precipitated "by any suitable reagent." In operating by acidification, the liquor is neutralised by purified lime or its carbonate, and the clear neutral liquor evaporated with addition of stearic acid and of small quantities of pure carbonate or stearate of barium, which react with the calcium sulphate to separate the volatile acids and other impurities. In order to bleach more completely, a dehydrogenerating substance, such as zinc powder, may be added.—A. H. A.

*Apparatus for Separating Impurities from Machinery Oil and Grease, and for Recovering Oil from Cleansing Waste, &c.* J. Davids, Manchester. Communicated from A. Koellner, of Neumuehlen, Germany. Eng. Pat. 4762, Oct. 6, 1883.

THE inventor claims the use of a hopper or chamber as a receiver and settling chamber for the dirty oil or grease; the application of warming or heating apparatus by means of which the grease or oil may be kept in a thin liquid state; perforated filtering chambers so arranged that the oil or grease has to pass from bottom to top, and then from top to bottom, automatically; a chamber in which the clean grease or oily material will gather, and from whence it can be taken at convenience for re-use; adjustable screws, whereby the filtering material in the perforated boxes can be tightened or loosened; and an air-pipe for permitting the free access or egress of air to and from the settling-chamber.—A. H. A.

*Obtaining Glycerine from Spent Lye produced in the Manufacture of Soap.* George Payne. Eng. Pat. 5257, Nov. 6, 1883.

THIS process consists in removing the small quantity of soap remaining in the lyes by precipitation with lime. After filtering, the excess of lime is neutralised with muriatic acid, the liquid evaporated, and the glycerine distilled in the well-known manner.—C. F. C.

*Refining and Deodorizing Oils and Fats.* Edgar Siddons Wilson. Eng. Pat. 5340, Nov. 12, 1883.

THE oils or fats to be treated are first made into a soap by treatment with caustic soda, and are then treated with chlorine or an alkaline hypochloride. The material treated can, if desirable, be obtained in the form of fatty acid by decomposing the decolorized soap with an acid.—C. F. C.

*An Improved Process for the Manufacture of Linseed Oil and Apparatus therefor.* Henry Alexander Davidson. Communicated by William Robert Lake. Eng. Pat. 6627, April 21, 1884.

THE object of this invention is to provide the means for the extraction of linseed oil, whereby more oil can be made from a given quantity of ground or other seed, and of a better quality than that made in the ordinary way, "all of which will be more clearly hereinafter shown by reference to the accompanying drawing." The method consists essentially in expressing the oil in ordinary presses, contained in a chamber which can be heated to any required temperature by means of steam pipes.—C. F. C.

### XIII.—TANNING, LEATHER, GLUES, SIZES, AND RESINS.

*Extract of Mangrove Bark for Tanning.* By John Fisher, Matlock, Derby. Partly communicated by C. M. Allen, Singapore. Eng. Pat. 5311, Nov. 9th, 1883.

THE bark of the mangrove tree (*Rhizophora*) is crushed, and if necessary soaked in cold water to remove the salt, then boiled with water, best under pressure, and the extract evaporated to the consistency of treacle. The extract and method of preparing it are claimed.—J. M. H. M.

*Treating Leather for Driving Belts.* J. Paterson. Eng. Pat. 5879, Dec. 27th, 1883. Provisional Protection only.

UNWROUGHT wood, resin, and gnmthus (frankincense) are melted together in the proportion of 42lbs. of resin, and 70lbs. of gnmthus; after well mixing, 1 gallon of boiled or linseed oil, and 2lbs. of commercial indiarubber solution diluted to a liquid with "benzoline spirit" or bisulphide of carbon are added. The mixture is then thinned down with "benzoline spirit" till it is about the thickness of molasses at ordinary temperature. The leather is soaked in this for a quarter of an hour, washed in "benzoline spirit," and then dried.—H. R. P.

*Utilisation of Leather Scraps or Waste.* C. B. Warner, London. Eng. Pat. 5975, 31st Dec., 1883.

THE scraps are cleansed, and then softened by dilute glycerine, the surplus of which is squeezed out. The scraps are then soaked for some hours in a solution of casein. After draining, the mass is passed through a series of rollers, first corrugated and then plain, and at gradually diminishing distances apart. While passing through the rollers the casein is coagulated by a fine spray of weak alum solution or of hydrochloric, acetic, or other cheap acid. The sheet so produced is finally subjected to heavy hydraulic or other pressure, and then slowly dried. If it be required to make it more pliable, it is sponged with glycerine and passed through rollers. The casein solution is prepared by boiling cheese or casein with 10 or 12 volumes of water, and afterwards adding sufficient caustic soda to complete the solution, which is cooled to 98° F. and strained. Instead of soda, liquid ammonia, or slacked lime may be used to complete the solution; in which case a temperature of 120-130° F. only is required. A small quantity of silicate of soda may be added.—H. R. P.

*Bleaching Leather or Tanned Hides.* G. W. Von Nawrocki. Communicated by G. Levinstein, Wiesbaden. Eng. Pat. 5965, 31st Dec., 1883. Provisional Protection only.

THE leather is impregnated with a soluble chemical compound, and then passed through a bath of some other solution, by which the first is precipitated. This treatment is repeated as often as required, and the leather is finally rinsed out in water. Chlorides or sulphates of zinc, or aluminium, or acetate of lead, fixed by passing through soda solution; or chlorides of barium or calcium, or acetate of lead, fixed by sulphuric acid, or aluminium sulphate, are mentioned as suitable.—H. R. P.

*Improvements in Tanning Hides and Skins.* A. M. Clark. Communicated by Wm. Maynard, of New York. Eng. Pat. 7933, 19th May, 1884.

A liquor is obtained from spent bark and other tanning material, by passing aqueous sulphurous acid through the tanks or "leaches" containing it; and this solution is used mixed with other weak liquor, for colouring and tanning green hides or skins, and also in the after operations, the quantity of sulphurous acid being increased in proportion to the density of the liquor.—H. R. P.

## XIV.—AGRICULTURE, MANURES, Etc.

*Influence of Hydroxylamine Salts upon Plants.* By V. Meyer and E. Schulze. Berl. Ber., 17, 1554.

THE supply of nitrogen to plants, whether natural or artificial, takes the form of nitric acid or ammonia, neither of which compounds possesses any synthetical activity. Hydroxylamine, occupying in regard to oxidation a position intermediate between these compounds, has on the other hand a synthetical activity of a high order, more especially in relation to organic oxy-compounds, such as the aldehydes and ketones. Moreover, the tendency to synthetical combination with such compounds is in many cases as well marked in its salts as in the free base. The authors therefore considered it highly probable, *a priori*, that hydroxylamine and its salts play an important part in the synthesis of amides and proteid substances in plants. With the object of testing this hypothesis, they investigated the growth of maize and barley under the condition of a supply of nitrogen in the form of hydroxylamine. Their experiments, which are fully described, included a comparison, under parallel conditions, with the same plants, fed with the N-equivalent quantities of ammonia and nitric acid. The result was to show that the hydroxylamine salts act as direct poisons. Indeed, they were found to possess powerful antiseptic properties; solutions of gelatine and hay extracts, to which small quantities of these salts were added, were found to be sterilised. In conclusion, the authors show that their hypothesis is not negated by the result; and that the possible functions of hydroxylamine remain to be investigated by means of experiments which more nearly attain to the conditions of the possible supply or existence of this base in the plant.—C. F. C.

*Improvements in the Construction of Silos, etc.* By Fredk. Lindsay. Leadenhall St., London. Eng. Pat. 5037, October 23rd, 1883.

DOUBLE walls of plain or corrugated iron are bolted to an iron or wooden frame. The inner wall is made air-tight by double riveting on two corrugations, the joint being made with red lead and yarn. The lower ends of the frame and inner walls are set in a concrete floor. Between the inner and outer walls there is a free circulation of air. One or both walls may be felted if required.—J. M. H. M.

*Manufacture of Manure from Sewage.* J. H. Kidd. Eng. Pat. 5550, Nov. 27th, 1883.

DRIED sewage-sludge or excretal matter is moistened with sulphuric acid, and placed in trays in a gas purifier, to absorb ammonia, either from gas, or from the distillation of ammoniacal liquor, sewage, or other sources.—H. R. P.

*Improvements in Construction of Silos.* By William Ellis, Chagford, Devon. Eng. Pat. 5256, March 21st, 1884.

THE side walls and part of the gables of the silo are of sheet metal plates inserted in grooves in the parts which are inserted in the base or sill partly sunk below the ground. At their upper ends the parts are similarly fitted into the top frame carrying the principals of the roof. To the purlins secured to the principals are bolted the plates forming the gable roof. The roof and roof frame are divided into two or more sections capable of being fixed or moved independently, and the roofing plates of the sections conveniently abut on each other above the principals. The fodder to be stored is fed in through openings in the upper wooden part of the gables. These doors open outwards and may be lined with zinc. The claim is for a silo which may be readily erected and taken down, constructed substantially as above described.—J. M. H. M.

## XV.—SUGARS, GUMS, STARCHES, Etc.

*Rate of Oxidation of Sugars, Starch, etc., by Fehling's Solution.* By F. Urech. Berl. Ber. 17, 1539.

THE author has continued his researches (Ibid. 17, 495), upon the mutual reaction of the sugars and Fehling's solution at the ordinary temperature; the equivalent quantity of the latter being added to the sugar solution, and after allowing the reaction to proceed, the quantity remaining undecomposed was determined at successive intervals of time.

*I. Group.* Parallel series with (a) dextrose, (b) invert sugar, (c) milk-sugar, (d) mixture of (a) and (c) in equal quantities. The duration of the experiments is given in hours in the first column. The numbers under (e) are the arithmetical mean of (a) and (c.) Temp. 12-3°.

Hours	a	b	c	d	e
3	2.19	6.9	1.13	1.73	1.66
7	7.21	17.83	3.99	5.95	5.60
13	14.79	34.53	8.56	11.68	11.67
21	23.96	49.48	14.37	18.80	19.16
31	32.56	60.93	20.50	26.31	26.57
51	44.03	71.04	32.71	38.38	38.37
83	58.73	79.35	45.93	51.73	52.33
123	73.00	87.06	60.83	66.73	66.91
195	86.03	93.99	76.38	81.25	81.20
291	94.75	98.56	88.55	91.81	91.65
411	98.80	99.90	95.35	97.72	97.07
536	—	—	98.52	99.68	99.25

The coincidence of the numbers under (d) and (e) proves that the reaction is unaffected by mixture.

*II. Group.* Parallel series with (a) invert sugar, (b) artificial mixture of levulose and dextrose in equal quantity, (c) dextrose, (d) levulose; (e) contains the numbers for levulose calculated from (a) and (c), and (f) the numbers calculated from (b) and (c), according to the respective equations:  $2a - c = e$ ;  $2b - c = f$ .

Hours	a	b	c	d	e	f
4	6.51	6.44	2.64	11.35	10.44	10.24
7	12.62	12.41	5.54	20.63	19.70	19.28
13	24.47	24.20	11.10	38.92	37.84	37.30
21	38.62	37.19	18.63	59.02	58.61	55.75
31	50.87	49.21	26.78	74.82	74.96	71.64
51	64.54	63.25	40.91	87.62	88.17	86.59
83	75.77	74.76	57.14	93.38	94.40	92.38
123	83.66	82.95	72.11	95.52	95.21	93.79
195	91.73	91.81	86.12	96.67	97.34	97.50
291	96.84	95.18	91.90	—	—	—
411	98.57	98.01	97.73	—	—	—
536	99.66	99.16	98.93	—	—	—

The coincidence between the numbers under (a) and (b) affords a fresh proof that the inversion of saccharose consists in its resolution into equal parts of dextrose and levulose. The slight variations between (e) (f), the author explains by the variable action of the alkali, its action upon levulose being greater than upon dextrose and milk-sugar.

*III. Group.* Series with milk-sugar, and milk-sugar after treatment with boiling dilute acids (so-called invert milk-sugar). (1) Parallel series with (a) milk-sugar and (b) invert milk-sugar with equivalent quantities of Fehling's solution. Temp. 120°. (2) (c) and (d) as in (a) and (b) respectively, the Fehling solution being in excess.

Hours	a	b	c	d
4	1.63	1.94	7	4.27
7	2.85	3.78	21	14.24
13	7.04	8.25	79	43.79
21	12.28	13.48	123	59.24
51	29.50	31.28	195	74.00
83	43.17	46.25	291	86.41
158	59.47	64.41	—	—
230	73.74	79.68	—	—
291	81.34	87.43	—	—
411	90.10	95.11	—	—

The author discusses the bearing of these numbers upon the mode of resolution, *i.e.*, inversion of milk-sugar; reducing the question to the alternative conclusions that either inverted milk-sugar consists of a lactoglucose, reducing less rapidly than dextrose, together with galactose, or it is composed of dextrose together with galactose of a low reduction rate. He promises a further investigation of this point by means of artificial mixtures.—C. F. C.



*On the Birotary Phenomena of Glucose.* By F. Urech, Berl. Ber. 17, 1547.

THE following are the main results of a comparison of milk-sugar with glucose in regard to this phenomenon:—

(1). Milk-sugar. Concentration 5.28 in 100 cc.  
Initial rotation : End rotation  $\frac{8.55}{5.2} = 1.62 : 1$

(2). Glucose. Concentration 5.19 in 100 cc.  
Initial rotation : End rotation  $\frac{11.5}{5.52} = 2.08 : 1$

(3). Glucose. Concentration 3.02 in 100 cc.  
Initial rotation : End rotation  $\frac{6.96}{3.18} = 2.19 : 1$

(4). Glucose: Concentration 16.92 in 100 cc.  
Initial rotation : End rotation  $\frac{38.00}{18.32} = 2.07 : 1$

The progress of the phenomenon is shewn by the results of determinations in series—the time varying. The influence of variations in the proportion of water is seen to be slight. The influence of the temperature is seen in the results of series (5) (6) and (7):—

	Concentration.	Temp.	Mean of constants. log a.
(5)	15.24 100 cc. ....	1° .....	3.307
(6)	17.00 100 cc. ....	0.5° .....	3.286
(7)	10.05 100 cc. ....	3.7° .....	3.485

—C. F. C.

*Action of Sodium Hydrate (Solution) upon Invert-Sugar, Dextrose, and Milk-Sugar.* By F. Urech. Berl. Ber. 17, 1543.

THIS is a continuation of the author's investigation, the conditions of experiment being varied in regard to the quantity of sodium hydrate present in the solution.

*Group I.* This quantity is twice that present in the normal Fehling's solution. Parallel series with (1) invert sugar, (2) dextrose, (3) milk-sugar. Temp. 12° 5.

As previously stated (Ibid. 17, 495) the rate of decomposition is expressed by the equation  $\frac{du}{dt} = au$ , in which

the integrating constant  $a = \frac{\log \frac{u_0}{u}}{\log_e t}$

Hours.	1.	log a	2.	log a	3.	log a
72	41.74	3.915	28.13	3.563	20.37	3.500
120	63.80	3.927	45.24	3.700	28.33	3.413
216	76.67	3.828	63.08	3.671	50.37	3.511
336	81.25	3.710	75.36	3.620	57.00	3.400
475	90.40	3.691	88.50	3.658	79.19	3.519
601	93.00	3.616	92.31	3.730	86.56	3.523
741	—	—	97.00	3.763	91.10	3.518

*Group II.* The quantity of sodium hydrate was normal. Temp. 12°. In other respects the same as in Group I.

Hours.	1.	log a	2.	log a	3.	log a
123	51.55	3.807	33.33	3.518	32.60	3.506
218	71.46	3.759	61.29	3.674	43.83	3.422
293	78.72	3.723	72.22	3.640	58.52	3.477
365	82.61	3.680	81.82	3.669	63.31	3.558
458	87.50	3.687	85.29	3.622	80.71	3.556

*Group III.* The quantity of sodium hydrate three times the normal. Temp. 12° 5. In other respects as in Groups I and II.

Hours.			Dextrose only.		
1	0.77	3.807	hrs.		
4.5	8.84	2.313	62.5	45.19	3.998
11.5	20.00	2.187	86.25	52.11	3.931
21.0	29.60	2.165	120.5	63.25	3.919
39.5	35.96	2.049	117	72.88	3.948
52.75	39.61	3.975	195.25	80.95	3.929

The author has also investigated the action of potassium hydrate upon these sugars. In the case of dextrose the alkali was employed in the ratio (molecular).  $\text{KOH} : \text{H}_2\text{O} : \text{C}_6\text{H}_{12}\text{O}_6 = 32.24 : 11.89 : 1$ . The mean results differed but slightly from those obtained with soda. The time required to render 50 per cent. of the sugar incapable of reducing Fehling's solution was 251 hours in the case of the latter, 231 hours with potassium hydrate.—C.F.C.

*On the Action of Cupric Hydrate on different kinds of Sugars.* J. Habermann and M. Hönig. Monatshefte für Chemie, 5, 208.

THE action of cupric hydrate on neutral and alkaline solutions of dextrose, levulose, saccharose and inverted sugar has already been described by the authors. (Monatshefte 3, 651.) The present paper relates to the action of the hydrate on similar solutions of galactose, milk sugar, maltose, sorbin, dulcitol, and mannite. For the mode of operation, reference is made to the previous paper.

*Galactose.*—Prepared from milk sugar by Loxhlet's method (Journ. f. pr. Chem. N. Folge 21, 269.) The formation of dextrose as a second decomposition product of milk sugar was fully confirmed. As in all previous cases, the reaction with cupric hydrate, as regards the nature of the oxidation products, was similar in both neutral and alkaline solutions, and varied only with the duration of the oxidation. The oxidation products distinguished by the authors were—(1) carbon dioxide; (2) formic acid; (3) glycollic acid in very small quantities; (4) relatively large quantities of lactic acid; (5) an acid, or mixture of acids, probably monobasic and polyatomic, and with four to six atoms of carbon. Similar products were formed from the sugars mentioned in the previous paper; (6) a residue of unaltered sugar. The solution was coloured more rapidly in alkaline than in neutral solutions. After boiling for half an hour the colour was a yellow-green; after an hour a pure reddish yellow. The evolution of carbon dioxide was slow at first, then, after addition of more cupric hydrate, it became very copious, but soon decreased again—the reaction generally was similar to that of grape sugar, but more rapid.

*Milk Sugar.*—The course of the reaction resembled that of cane sugar, and in this case also inversion preceded oxidation. The oxidation products resembled those of galactose, but milk sugar yielded larger quantities of glycollic acid, but not so much lactic acid.

*Maltose.*—Prepared by Herzfeld's method. (Annalen 220, 209.) The cupric hydrate was reduced with great rapidity, especially in alkaline solutions. The reaction, as usual, took place in three stages; in the first, during which reduction was slow, inversion undoubtedly occurred. The decomposition products resembled those of dextrose, both qualitatively and quantitatively.

*Sorbin.*—The substance was obtained from H. Trommsdorff in Erfurt, and was nearly pure. The reaction with cupric hydrate was distinguished by its energy and rapidity. The solution assumed a yellow-red colour after boiling from ten to fifteen minutes, but even in this case the evolution of carbon dioxide was slow at first, becoming very rapid at a later stage. With alkaline solutions the liquid was of a blue-green colour at the end of the reaction, but became colourless when warmed, so that probably intermediate products with powerful reducing properties were formed. The whole of the sorbin was always reduced, and the oxidation products were—(1) very large quantities of carbon dioxide; (2) a considerable amount of formic acid; (3) a very small quantity of a brown substance, which could only be partially neutralised with metallic carbonates, and which was found to be a mixture of a neutral resinous substance with an acid which formed an amorphous barium salt; (4) an acid of the same composition as glyceric acid,  $\text{C}_3\text{H}_5\text{O}_4$ . The properties of the barium and potassium salts agreed in most respects with those of the salts of glyceric acid, but the authors do not consider the identity of the acids as proved with certainty.

*Mannite and Dulcite.*—Cupric hydrate has no action on these substances, either in alkaline or neutral solution, as might be expected from their inability to reduce Fehling's solution. The authors conclude that the formation of the above-mentioned oxidation products can be easily explained, by considering glucose, levulose and sorbin as pentatomic aldehyde and ketone alcohols respectively, and the probable formation of glyceric acid from sorbin is thus intelligible. On the other hand, the production of lactic acid from galactose and milk sugar is difficult to understand, if these bodies are supposed to possess an analogous constitution.—S. Y.

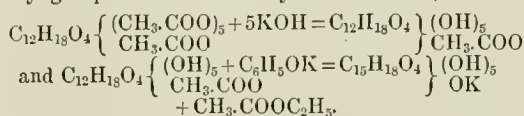
*On the Composition of the Unfermentable Substance existing in Commercial Starch Sugar.* C. Schmitt and A. Cobenzl. Berl. Ber. 17, 1000.

THE existence of an unfermentable substance in starch sugar has been noticed by several observers, but no definite conclusion has been arrived at as to its chemical nature, and the accounts given of its physiological action are very contradictory. According to the author and Neubauer, the substance has not the nature of a gum, nor is it a sugar. The substance was prepared by the authors by causing five kilos. of pure grape sugar, obtained from Messrs. Reny and Wahl, to ferment by means of yeast. The fermentation of the solution, the temperature of which was kept at 18° to 20°, and which contained twenty per cent. of sugar, was completed in five to six days. It was then filtered, evaporated as far as possible, and shaken vigorously in a large flask with a great excess of absolute alcohol. The alcohol was poured off, and the residue shaken with a fresh quantity. By repeating this process several times the unaltered sugar and other impurities were removed, and the syrup was converted into a yellowish grey mass. It was transferred to a mortar, and triturated with a mixture of equal quantities of alcohol and ether, when it was obtained in the form of a grey powder. It was filtered quickly with the pump, and dried over sulphuric acid. It was purified by dissolving in water, boiling with freshly-ignited animal charcoal, filtering and precipitating with alcohol and ether. It was thus prepared as a pure white powder, which was filtered and dried as before. The authors propose the name Gallisin for the compound. It is amorphous, and more hygroscopic than calcium chloride. It is insoluble in ether, chloroform, and hydrocarbons, and almost insoluble in absolute alcohol, but more soluble in methyl-alcohol and glacial acetic acid. It dissolves in a boiling mixture of equal parts of alcohol and glacial acetic acid. The concentrated aqueous solution has a distinctly acid reaction. Its relations to the sugars, carbo-hydrates and dextrins are shown by the following observations. It is unaltered, even when warmed, and after standing for a long time, by lead acetate, mercuric chloride, mercuric nitrate, ferric chloride, tincture of iodine, chloride of calcium, and chloride of barium. It forms a slight white precipitate, with concentrated solutions of barium hydrate, especially on addition of alcohol. It reduces silver nitrate with separation of silver; there is no action at first, but it occurs when the solution is warmed, and more rapidly on addition of alcohol. Potassium permanganate and bichromate are rapidly reduced by the substance, the former in a weak alkaline solution, with precipitation of manganese dioxide, the latter in a dilute sulphuric acid solution, with evolution of gas. It reduces both Fehling's and Knapp's solutions, and it was found that 0.05grm. glucose has the same reducing power as 0.109784grm. gallisin. The presence of gallisin prevents the precipitation of iron with ammonia or alkalis. An acetyl derivative is formed by the action of acetic anhydride on the compound. When treated with dilute mineral acids or oxalic acid on the water bath, gallisin is converted into grape sugar. Gallisin does not ferment, either with yeast or the ferment of cheese, but mould forms rapidly when its dilute aqueous solution is allowed to stand. Gallisin loses water and carbon dioxide below 100°, the mass frothing up. The substance has a sweet taste at first, but it soon becomes insipid. The results of three analyses,

in which great care was taken to avoid contact with air, established the formula  $C_{12}H_{22}O_{10}$ . The optical behaviour of gallisin was studied by the author, who employed this recently-described method of Landolt, but he was unable to obtain sufficiently accurate data to determine definitely the specific rotatory power of the substance. The results obtained show, however, that gallisin is dextro-rotatory, and appear to indicate that the rotatory power increases as the solution becomes

more dilute. Employing the equation  $\alpha_j = \frac{\alpha}{p \cdot l}$ , where  $\alpha$  = deviation,  $p$  = strength of solution in grms. per cc.; and  $l$  = length of tube in decimetres, the following numbers were obtained with solutions of decreasing strength.  $\alpha_j = 77.32^\circ$ ;  $80.10^\circ$ ,  $82.33^\circ$ ,  $82.76^\circ$ . Gallisin-barium was obtained by treating a concentrated aqueous solution of gallisin with an alcoholic solution of barium hydrate as a white flocculent precipitate. It is not easy to obtain the salt pure, for it decomposes readily with absorption of carbon dioxide. It is easily soluble in water, and the solution has an alkaline reaction, and quickly becomes turbid from formation of barium carbonate; it reduces Fehling's and Knapp's solutions. The salt has the composition  $C_{12}H_{22}BaO_{10} + 3H_2O$ . Hexacetyl gallisin was prepared by heating gallisin with three times its weight of acetic anhydride in sealed tubes to 130° to 140° for two to three hours. The substance is precipitated on addition of water, but not alcohol, hence the acetyl compound could be separated from unaltered gallisin by treatment with alcohol. The alcoholic solution was evaporated on the water bath to remove anhydride, redissolved in alcohol, decolourised with animal charcoal, and evaporated in the desiccator. It was thus obtained as a colourless glassy mass, the composition of which was found to be  $C_{12}H_{18}O_4(C_2H_3O_2)_6$ . It is insoluble in water, but dissolves easily in alcohol, ether, chloroform, carbon bisulphide and benzene. With excess of acetic anhydride at 170° to 180°, the amount of the acetyl compound was much smaller, and acetic ether was formed in large quantity.

When solutions of hexacetyl gallisin and caustic potash in absolute alcohol were mixed, a yellowish precipitate was formed at first, which re-dissolved on shaking, but was re-precipitated on addition of sufficient alkali to give a slight alkaline reaction. The substance was washed with alcohol and ether, and dried in the desiccator. It is less hygroscopic than gallisin, but dissolves easily in water, the solution being distinctly alkaline. The substance has the formula  $C_{12}H_{22}KO_{10}$ , so that five of the acetyl groups behave differently from the sixth, thus—



The lead salt was obtained by treating a concentrated aqueous solution of the potassium salt with an alcohol in solution of lead acetate in excess of alcohol, as a white flocculent precipitate. It is not very hygroscopic, but is easily soluble in water and in an alcoholic solution of lead acetate, but is re-precipitated on addition of a little basic lead acetate. The composition of gallisin lead is  $C_{12}H_{22}PbO_{10} + PbO$ .

*Conversion of Gallisin into Grape Sugar.* Neubauer observed that the unfermentable substance was formed by the action of acids on starch, and that by this same action it was eventually converted into grape sugar.

The author heated the syrup obtained after the fermentation of grape sugar with an equal weight of oxalic acid to 103° to 104° for two to three hours, and thus obtained a quantity of pure glucose. The reaction was found, however, not to be quantitative, for after the gallisin had been heated with a certain quantity of oxalic acid, and when the conversion was still incomplete, the solution turned yellow, and smelt strongly of caramel. The oxidation of gallisin was effected by adding 150cc. of an aqueous solution containing 100grms. of the substance to 400grms. of concentrated nitric acid in a large flask. A violent reaction took place at once, with evolution of red fumes, and the liquid began to boil. After a time



the solution was gently warmed until the evolution of red fumes had ceased; it was then allowed to cool, filtered from oxalic acid, made slightly alkaline with concentrated caustic potash, and then strongly acid with acetic acid. Beautiful white needles of an acid potassium salt were thus obtained. The solution was poured off, the crystals filtered with the pump, washed with cold water, and re-crystallised from hot water. The yield was 20 per cent. of the gallisin employed. The aqueous solution had an acid reaction. On analysis the composition of the substance was found to be  $C_6H_8K_2O_8$ . The normal silver salt was prepared by treating the aqueous solution of the potassium salt with silver nitrate; it was thus obtained as a white flakey precipitate, which became crystalline on warming, but soon turned black. Its composition proved to be  $C_6H_8Ag_2O_8$ . The free acid was prepared from the lead salt by decomposing it, while suspended in water, with sulphuretted hydrogen. It formed after evaporation a thick, strongly acid syrup, easily soluble in water and alcohol, but insoluble in ether. The ammonium salt, when distilled, yielded pyrrol and other empyrenumatic vapours, and a sublimate soluble in water, alcohol, and ether, hence the substance is probably saccharic or an isomeric acid.

When gallisin was heated in a current of hydrogen, it lost 4 molecules of water and one of carbon dioxide, and the glassy residue resembled gallisin closely. It formed a barium salt of the composition  $(C_{11}H_{23}O_8)_2Ba$ , so that the water evolved on heating was taken up again on solution.

*Detection of Gallisin in Sweetened Wine.*—A large quantity of a wine of known composition was evaporated on the water bath, and the red-brown residue treated with excess of ordinary methyl-alcohol. The solution after purification with animal charcoal was treated with a large amount of ethyl-alcohol, when a white flocculent precipitate was obtained. This was washed with alcohol, dissolved in a little water, and the solution added to a mixture of equal parts of absolute alcohol and ether. A white powdery precipitate of gallisin was thus obtained, the formula of which was found on analysis to be  $C_{12}H_{22}O_{10}$ . The barium salt was also prepared.

A large number of experiments made by Dr. Pfeiffer on the physiological action of gallisin on both men and animals showed that the substance has no injurious action whatever, and the authors therefore point out the injustice of the punishment of persons for adding starch sugar to wines in cases where no deception was intended.

S. Y.

## VIII.—SANITARY CHEMISTRY, DISINFECTANTS.

*On the Existence of Lead in Water passing through Lead Pipes.* C. Schneider. Arch. Pharm. 22, 185 to 192.

THE author draws the following conclusions from his experiments:—(1.) The soft water of the river Bober, which contains traces of sulphuric acid and of calcium salts, is capable of taking up considerable quantities of lead from new pipes. (2.) The formation on the inner surface of the pipes of a difficultly soluble protecting layer does not take place quickly. (3.) Even after 18 years' use lead pipes are not so much altered that water is quite prevented from dissolving the lead. (4.) Hard water containing 10.57 grms. sulphuric acid and 11.2 grms. lime per hectolitre is capable of dissolving lead. The author considers that the presence of carbon dioxide, even in small quantities, is sufficient to explain the solvent action of water, and disapproves of the use of lead pipes for spring waters.—S. Y.

*Preparation for destroying Disease Bacteria.* By Edward Sonstadt, Cheshunt, Herts. Eng. Pat. 4920, October 16, 1883.

CALCIUM iodate solution, which is inimical to bacterial life, is rendered mould-proof by addition of an alkaline salicylate. To increase the solubility of the calcium iodate it is dissolved not in pure water, but in a saturated

solution of sodium or potassium citrate. The saturated mould-proof solution of calcium iodate thus obtained is employed for inhalation, for dressing sores and wounds, for injections, &c. For certain purposes quinoline tartrate and sometimes glycerine also are combined with the above materials. Dry preparations containing the same ingredients are also claimed.—J. M. H. M.

## XIX.—PAPER, PASTEBOARD, Etc.

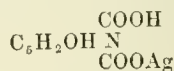
*Improvements in Machines for making Woody Fibre.* Herbert John Haddan. A communication from Philip H. Holmes, of Gardiner, U.S.A. Eng. Pat. 4784, March 12, 1884.

THIS invention relates to machinery for preparing wood fibre by supporting and rotating a block of wood against a knife arranged to cut and sever the fibre by a drawing motion. Also to the means of sharpening the knives or cutters while the machine is in operation, and thus retaining a straight and sharp edge on the cutters for a length of time. As fibre of different lengths may be desired for different purposes, each machine may be provided with a set of knives, so that simply by changing the knives any desired length of fibre may be produced. The knives are fed forward to the work, which is one of the important features of the improvement. They are at the same time reciprocated so as to produce a drawing cut.—H. A. R.

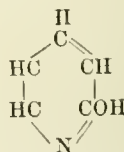
## XX—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Some Derivatives of Pyridine* by W. Königs and R. Geigy. Berl. Ber. 17, 589.

*Oxyppyridine-monocarbonic Acid* is formed on heating oxyquinolic acid with water at 195°. It crystallises in needles, which are anhydrous; it may be sublimed if carefully heated. It is different from the three acids of the same empirical composition hitherto known (J. Pr. Chem. [2], 27, 289 and 29, 16). *Oxyppyridine* is produced in the dry distillation of the mono-argentic oxyquinolate,



(Ber. 16, 2152); it crystallises in needles (m. p. 107°). In concentrated aqueous solution it reacts with bromine to form the dibromocompound,  $C_6H_4Br_2NO$ , which crystallises in long white needles (m. p. 207°). The formation of this oxyppyridine from quinolic acid is analogous to that of carbostyryl from quinquinic acid, whence we may infer for the former the analogous constitution represented by the formula



*Pyridinedisulphonic Acid* is formed by the action of sulphuric acid upon piperidine. It is distinguished from the monosulphonic acid by its reaction with bromine. The latter is easily converted into a dibromopyridine by adding bromine to its boiling aqueous solution; whereas the former is but very imperfectly converted into this compound by heating, at 160° to 180°, with three times its weight of bromine. On heating its barium salt with phosphorus penta-chloride at 200°, chlorinated derivatives of pyridine are formed. A trichloro pyridine may be isolated from them, and obtained, by crystallising, in plates (m. p. 48°).—C. F. C.

## XXI.—EXPLOSIVES, MATCHES, Etc.

*Improvements in Prismatic Powder for Heavy Ordnance.* Thorsten Nordenfelt. Eng. Pat. 4988, Oct. 19, 1883.

"At the present time it is common to mould powder intended for heavy ordnance into hexagonal or prismatic blocks, having flat ends. Now I form such blocks with convex ends in order that the surfaces may ignite with greater certainty at the first moment." It is also usual to provide these prismatic blocks with a perforation through their axis, with the object of offering at the moment of ignition a large surface to the flame, and of regulating the surface of combustion, for as the area of the exterior decreases by being burnt away, that of the interior increases by the enlargement of the hole. But a powder grain thus perforated is liable to be broken by the pressure of gas within it immediately after ignition, and the surface of combustion being thereby increased, there is the risk of an excessive and dangerous pressure being generated. "To avoid this risk, I substitute for the perforation, indentations on the two opposite surfaces of the grain, so that the first igniting surface is as large or nearly as large as when the grain is perforated, and the curvature of the surfaces favourable to regular ignition. The grains so formed are much less liable to be broken on ignition than are grains which are perforated in the usual way. As the grains burn away, the indentations presently meet, and after that moment the grain becomes a perforated grain, and operates in a similar manner. If the grain should break at this stage, in the latter stages of combustion no harm will result, as the projectile has then already proceeded a considerable distance along the bore, and the pressure behind it has fallen considerably." To effect the object of this invention, gunpowder is consolidated by hydraulic or other pressure in moulds corresponding in form to the blocks of powder described above.—W. D. B.

## XXII.—GENERAL ANALYTICAL CHEMISTRY.

*On the Separation of Strontium and Calcium.* Iwan Bogomoletz. Berl. Ber. 17, 1058.

THIS is a criticism of a paper bearing the same title, by Hr. Sidersky (Zeitschr. für Anal. Chem. 22, 10, and Berl. Ber. 16, 261). According to Sidersky, when a mixture of ammonium sulphate and ammonium oxalate is added to a warm neutral solution of the salts, the strontium is precipitated as sulphate, and the calcium as oxalate. The precipitate is washed and treated with dilute hydrochloric acid, when the calcium is dissolved and the strontium left behind. The calcium is then precipitated from the filtrate with ammonia. The method might be simplified by adding the ammonium salts to a warm acidified solution when the strontium only would be precipitated as sulphate. The method depends on the following observations: (1) When a mixture of ammonium oxalate and sulphate is added to a neutral strontium solution the precipitate consists entirely of strontium sulphate; (2) When the mixture is added to a neutral calcium solution the precipitate contains only calcium oxalate. Sidersky explains this by the greater affinity of strontium for sulphuric than for oxalic acid, and the reverse in the case of calcium, as indicated by the relative solubility of the salts. The author points out, however, that the oxalates of both strontium and calcium are less soluble than the sulphates, and that when strontium sulphate is treated with ammonium oxalate, a small quantity of strontium oxalate is formed. In this case there is a change from one insoluble salt into another, and it might be expected that the conditions which have been found by Dulong, Rose, Malagutti, Guldberg and Waage, and others, to hold in such cases, would apply to these also. 10 cc. of a 20 per cent. solution of strontium nitrate were treated at the ordinary temperature with 15cc. of a solution containing 20 per cent. of ammonium sulphate and 3 per cent. of ammonium oxalate; the precipitate contained some strontium oxalate. The experiment was repeated at 100°, when the precipitate was found to consist entirely of sulphate. The same quantity of strontium

nitrate was added to a warm mixture of 15cc. of the ammonium salts as above, and 90cc. of 3 per cent. ammonium oxalate solution; in this case a large quantity of strontium oxalate was found in the precipitate. When pure strontium oxalate was boiled for a few minutes with excess of a 20 per cent. solution of ammonium sulphate, the whole of the oxalate was converted into sulphate; on the other hand, when pure precipitated strontium sulphate was boiled with excess of a 4 per cent. solution of ammonium oxalate, a great part of the sulphate was converted into oxalate. Again, when pure calcium oxalate was boiled with excess of a strong (20-25 per cent.) solution of ammonium sulphate, the whole of it went into solution, owing to the formation of calcium sulphate. The oxalate was formed again by the addition of a large quantity of ammonium oxalate. The availability of Sidersky's method, therefore, depends on the conditions as regards mass, time and temperature under which the experiment is conducted. When the method is simplified by the addition of the mixture of ammonium salts to the acidified solution, it appears to be essentially similar to the ordinary method described by Rose (Poggendorff's Ann. 110, 296).—S. Y.

*Note on the Analysis of Volatile Organic Sulphur-Compounds.* Victor Meyer and Otto Stadler. Ber. 17 [11] 1576.

A KNOWLEDGE of the following remarkable observation may prevent much error and loss of time in analytical work:—The circumstance was noticed during the examination of a volatile oil, which was known to contain carbon, hydrogen and sulphur; it was also probable that it contained nitrogen, and a portion of the substance was therefore tested by melting with potassium, but on account of the large percentage of sulphur, it was impossible to decide by this method. A nitrogen determination was next made, according to Dumas' method, and a considerable volume of gas was collected, corresponding to about 14 per cent. of nitrogen; thus there appeared to be no doubt that the substance under examination was rich in nitrogen. But a close investigation of the body tended to throw some doubt upon the accuracy of the above experiment, especially as most volatile bodies undergo combustion very quickly. A second determination was therefore made; this was conducted more slowly, and to the astonishment of the authors only a few cubic centimetres of gas (corresponding to 3.1 per cent.) came off. This rendered a third experiment necessary; in this a thick layer of lead chromate was employed, and the substance was heated very gradually; the gas evolved was almost entirely absorbed by alkali, showing that the body was free from nitrogen. The gas collected in the earlier experiments was now tested; it burned with a blue flame, and proved to be carbon monoxide: a solution of cuprous chloride in HCl absorbed it almost without residue. These strange results must have been due to the large amount of sulphur contained in the substance, and the ease with which it volatilised. When the combustion was rapid, a considerable quantity of sulphurous acid was probably produced, which, under the given circumstances—presence of copper spirals, etc.—reduced the carbonic acid to carbon monoxide. Scarcely any carbon monoxide was obtained when substances free from sulphur were tried; but carbon monoxide is obtained when CO<sub>2</sub> and SO<sub>2</sub> are passed together over glowing copper spirals. These results show that in the analysis, according to Dumas' method, of substances containing both sulphur and nitrogen, the combustion should be carried on very slowly, and a thick layer of PbCrO<sub>4</sub> should be used. The gas collected should also be tested for carbon monoxide.—D. E. J.

*A Simple Method for the Determination of Halogens in the Side-Chains of Aromatic Compounds.* R. E. Schulze. Ber. 17 [12] 1675.

WHEN benzyl-chloride or benzal-chloride is heated to boiling with an alcoholic solution of silver nitrate, AgCl separates out; the method gives good analytical



results, and has been tested by the author in the case of the above-mentioned bodies, as well as of  $\beta$ -naphthyl-chloride and -bromide. The analysis is best carried out as follows:—A convenient quantity of the substance is weighed out into a small flask, an excess of a hot saturated solution of  $\text{AgNO}_3$  in alcohol is added, and the whole is boiled for five minutes in connection with a reversed condenser; care should be taken that the tube of the latter reaches well down into the neck of the flask, so that the cork may not come into contact with the liquid. The silver salt is collected on one of the well-known "platinum filters," covered on the bottom with a thin layer of fine asbestos, and fitted by means of an indiarubber band to a funnel and suction apparatus. The asbestos is first moistened with alcohol, and then the contents of the flask are poured in; the precipitate is washed, first with alcohol, then with water acidulated with  $\text{HNO}_3$ , and lastly again with alcohol; the precipitate may then be heated gently over the naked flame. The whole analysis takes up at most half an hour. The method has this advantage, that any halogen contained in the aromatic nucleus is not attacked. The precipitate is not contaminated with nitro-products, as often happens in Carius's method; indeed, in the case of compounds, such as  $\beta$ -naphthyl-chloride and -bromide, it is impossible to use Carius's method on this account. The author invites chemists to assist him in working out this method of analysis.—D. E. J.

*Analytical Notes on Hydrocyanic Acid.* By Louis Siebold. Read before the Pharmaceutical Conference, 1884.

THE author refers to the serious error, previously pointed out by him, resulting from the use of an insufficient amount of alkali in the volumetric estimation of hydrocyanic acid by Liebig's process, and criticises the suggestions made by Dr. Senier and Mr. MacEwan, with the object of avoiding this error. As the safest means of obtaining perfectly trustworthy results, he recommends a first or preliminary titration to be made in the presence of a very decided excess of standard normal soda solution, and having thus approximately ascertained the amount of hydrocyanic acid present, and by calculation also that of the standard soda required, and a second estimation then to be made in the presence of only a slight excess of alkali over and above the proportion found in the first experiment to be needed for the conversion of the acid into sodium cyanide. In this manner a scientifically exact result is obtained. With regard to the process of the United States Pharmacopœia, the author confirms his statement made at last year's meeting of the Conference as to the accuracy of this method, but insists on the necessity of this titration being performed rapidly, so as to prevent loss of  $\text{HCy}$  by evaporation during the process. It is best to run the acid from the pipette on the excess of magnesia and a small quantity of indicator (potassium chromate) contained in the beaker, then to add the silver solution from the burette in a thin stream until the end is nearly reached, and then to complete the reaction by adding the remainder of the silver solution drop by drop. He further shows that, if the same precautions be adopted, the carbonates of magnesium, calcium, and barium can be substituted in this process for the magnesium oxide, and that this holds true, not only of the carbonates obtained by precipitation, but also of the corresponding native minerals, such as calc-spar, magnesite, etc. The author does not recommend these substances to be used in the place of magnesia, but merely wishes to record as an interesting fact that a body of such extremely weak acid properties as hydrocyanic acid, which by itself is without action on these minerals, can be made to decompose them completely, and even without heat, by the intervention of silver nitrate. In conclusion, he again lays stress on the absence of hydrochloric acid and chlorides as an absolute condition in the American process of estimating this acid.

*Phenol-phthalëin as an Indicator in the Estimation of Carbon Dioxide in Mixtures of Gases.* R. Blochmann. Berl. Ber. 17, 1017.

PHENOL-PHTHALEIN was first proposed as an indicator by E. Luck (Fresenius, Zeitschr. für anal. Chem. 16 (1877), 332. It cannot be used in presence of ammonia, but is the best indicator in the estimation of pure alkalis and alkaline earths. With alkaline carbonates reliable results can only be obtained on heating, for in the cold decolorisation takes place as soon as the bicarbonate is formed. The disappearance of the colour does not, however, take place at once, and hence the estimation of mixtures of carbonates with caustic alkalis by this method is not very reliable. The power of free carbon dioxide to decolorise phenol-phthalëin may, however, be made use of in the estimation of that gas when mixed with others. When 1 to 2 cc. of lime water and a few drops of phenol-phthalëin are shaken in a test tube filled with exhaled air, the colour soon disappears. Even when the carbon dioxide is still more diluted, as in ordinary air, the indicator is available. A little distilled water is boiled in a platinum basin, and three drops of a solution of pure phenol-phthalëin in 60 per cent. alcohol (1:1000) is added. The solution is then treated drop by drop with lime water until it is distinctly coloured. On shaking this solution vigorously in a half-litre flask filled with ordinary air the colour disappears unless there is an excess of lime water. The colour is at once restored by the addition of an alkali. In estimating the carbon dioxide present in the air, the best results seem to be obtained when the volume of air required to neutralise a known quantity of lime water is measured, the point of saturation being indicated by the disappearance of the colour. Lux has proposed to employ the colouring matter called Flavescëin, which is obtained by the distillation of oak-wood with superheated steam for the same purpose (Zeitschr. für anal. Chem. 19 (1880), 467), but the author has not tested its value. Degener's Phenacetolin does not answer the purpose. The proposal of Lux appears not to have been generally noticed, for a more recent method by Woolpert (Centralblatt für allgem. Gesundheitspflege, II., Jahrgang (1882) depends on the appearance of a turbidity which is much less easy to recognise with certainty than the disappearance of a colour. Angus Smith (Air and Rain, 1872, p. 193) long ago called attention to the want of a good indicator, and pointed out that neither litmus nor rosolic acid could be employed.—S. Y.

*On a Reaction for Hydrogen Peroxide.* Moritz Traube. Berl. Ber. 17, 1062.

SCHÖNBEIN's delicate and characteristic test for hydrogen peroxide, in which potassium or zinc iodide, starch and ferrous sulphate are the reagents, is only available in neutral solutions. In strongly acid solutions, when but little peroxide is present, no colouration at all takes place. The author finds that in very acid solutions the reaction is perfectly reliable in presence of a small quantity of copper sulphate. If some sulphuric acid, zinc iodide and starch are added to 6 or 8 cc. of a solution containing only traces of hydrogen peroxide, then 1 to 4 drops, but not more, of a 2 per cent. solution of copper sulphate, and lastly a little  $\frac{1}{2}$  per cent. solution of ferrous sulphate, a blue colouration is produced at once or after a few seconds. In this way the loss of time involved in neutralising the solution is avoided, as well as the possible decomposition of peroxide by the heating of the solution, and the decrease of sensitiveness by its dilution. The reaction is available in solutions consisting of two parts of water and one of concentrated sulphuric acid. If the acid is stronger the iodide itself is decomposed. Alkaline solutions should be acidified with dilute sulphuric acid. It is to be observed that the ferrous sulphate must be added after and not before the copper sulphate. The reaction depends on the fact that in very dilute solutions soluble cupric iodide is formed without liberation of iodine (M. Traube, Berl. Ber. 17, 1064), and the latter is only set free in presence of hydrogen peroxide and ferrous sulphate. Of course it is

necessary to add only a very small quantity of copper sulphate, or iodine would be liberated and cuprous iodide formed.—S. Y.

### *On the Oxidation and Estimation of Chromium Oxide.* H. Baubigny. Bull. Soc. Chim., 41, 291-301.

THE presence of chromium oxide sometimes causes and sometimes prevents the precipitation of other oxides, and it is therefore customary to oxidise the chromic oxide to chromic acid. This is best effected as proposed by Storer (Proc. Amer. Acad. Arts. Sci. 1859, 6, 338) and Pawolleck (Berl. Ber. 16, 3008), by heating the solution with nitric acid and potassium chlorate. The oxidation takes place at once even when the oxide is not in solution, is in the case of iron ore; and the strength of the acid need not be 39 B. as stated by Pearson (Silliman's Journ. (2) 48, 190). The oxides should not be precipitated with ammonia, for the chromic acid may be partially reduced by the products formed by the action of the chlorous acid on ammonia; it is therefore better to add a slight excess of sodium bicarbonate. The precipitated alumina contains only traces of chromium after being washed with a dilute solution of bicarbonate. The chromium is precipitated from the hot alkaline filtrate by means of ammonium sulphide, and it must be washed free from alkali, or it may be oxidised when ignited, and chromic acid may be volatilised. If the chromium is estimated as chromic acid it is best to convert it into the lead salt in a dilute solution of acetic acid. Its precipitation as mercurous chromate is not advisable in presence of ammonium salts, for partial reduction may take place.—S. Y.

## Monthly Patent List.

COMPILED BY WM. P. THOMPSON AND CO.

### ENGLISH APPLICATIONS.

1881.

- 10424 A. de Maroussem, Brussels. An improved process of refining sugar. Complete specification, July 22.  
10427 F. W. E. Shrivell, London. Liquid hop auxiliary. Complete specification, July 22.  
10431 G. F. Busbridge and J. H. Turvey, East Malling, Kent. Improvements in apparatus for the manufacture of cement. July 22.  
10437 L. A. Groth, London. A new or improved apparatus for injecting and mixing fluid carbonic acid in small regulated quantities with beer and sparkling liquors. Communicated by Dr. E. Lühmann, Rogasen, Germany. Complete specification, July 22.  
10438 L. A. Groth, London. A new or improved apparatus for and method of preparing textile fibres and fabrics by the help of fluids. Communicated by H. Giesler, Molsheim, Germany. July 22.  
10439 L. A. Groth, London. A new or improved apparatus for and method of chemically treating wool, cotton, and other soft goods. Communicated by H. Giesler, Molsheim, Germany. July 22.  
10440 L. A. Groth, London. A new method of preserving hops. Communicated by J. A. Gfahl, Innsbruck, Tyrol. July 22.  
10448 R. Main, Ardeer, and W. Galbraith, Glangarnock, Ayrshire. Improvements in the process of obtaining ammonia or like products from the gases evolved from blast furnaces or from the gases produced from the destructive distillation of coal, shale, or other carbonaceous substances, and in the means employed therefor. Complete specification, July 22.  
10457 J. Pickering, London, and H. E. A. Saale, Chiswick. An improved anti-fouling composition for coating ships' bottoms and submerged metal structures. July 22.  
10459 H. Porter, Honour Oak, Surrey, and J. H. Porter, London. Improvements in apparatus or means to be employed in effecting the softening or purification of water, and for other purposes. July 22.  
10482 J. Hanson, Bingley, Yorkshire. Improvements in apparatus for purifying coal gas. July 23.  
10490 R. Stone, London. Improvements in the manufacture of plaster from chalk, lime, gypsum, or such like materials, and in machinery and appliances for carrying same into effect. January 1. Previously included in No. 153.  
10495 E. Capitaine, London. Improvements in the treatment of the lupinus plant (Lupine) to adapt it for use as food for cattle. Communicated by H. Habermann, Berlin. July 23.  
10523 E. W. R. Schröter, Hamburg, Germany. A new medical compound, and the process of manufacturing the same. July 24.

- 10528 J. A. R. Main and J. Dick, Glasgow. Improvements in apparatus for drying and heating air. May 26. Previously included in No. 8195.  
10571 C. Thompson, Sunderland. Improvement in apparatus employed in the manufacture of malleable iron and steel. Complete specification. July 25.  
10600 S. Pitt, Sutton. Improvements in the manufacture of waterproof or impermeable fabrics. Communicated by The Company for Manufacturing Waterproof Fabrics, St. Petersburg, Russia. July 25.  
10601 C. R. A. Wright, D.Sc., London. Improvements in the method of shaping soap into bars. July 25.  
10620 T. Froggatt, sen., Cheltenham. Steeling iron without welding or blistering. July 26.  
10643 H. W. Lee, Westminster. Method of separating solid and liquid fecal matters and appliances for that purpose. Communicated by T. Roberts, Christchurch, New Zealand. July 26.  
10648 J. C. Stephenson, Landport. Improvements in the preparation of a material for deodorising purposes, and for use in the purification of oil syrups and other liquids. Complete specification. July 26.  
10651 J. Mactear, Glasgow. Improvements in making granulated crystalline carbonate of soda and in apparatus therefor. July 28.  
10675 E. G. Brewer, London. A new or improved mode of and apparatus for the carburation of gas in railway and other carriage lamps and lanterns. Communicated by J. E. Dery, Brussels. July 28.  
10686 A. M. Clark, London. An improved method of converting the juice of the Jerusalem artichoke into levulose, and the application of the latter for obtaining various products by fermentation and distillation. Communicated by E. L. J. Boniface, Paris. July 28.  
10697 W. Charleson, Leigh, Lancashire. Grease-proving composition. July 29.  
10730 W. McDonnell, Dublin. A new product for enriching milk or making artificial cream, and a process for making such product. July 29.  
10739 M. Gehre, Hanover, Germany. Method and means for recovering and utilising the heat of exhaust vapours or gases. July 29.  
10740 W. R. Lake, London. Improvements in apparatus for purifying and maturing liquors or distilled spirits. Communicated by the Cushing Process Company, Incorporated, Boston, U.S.A. Complete specification. July 29.  
10743 G. F. Redfern, London. A new or improved process for extracting sulphur from sulphide of hydrogen. Communicated by Dr. H. von Miller, and C. Opl, Hruschau, Austria. Complete specification. July 29.  
10765 L. P. Merriam, Homerton. Improvements in the manufacture of artificial ivory. July 30.  
10786 C. D. Abel, London. An improved manufacture of toughened opal glass articles and apparatus therefor. Communicated by La Compagnie Générale du Verre et du Cristal Trempe, Paris. July 30.  
10790 J. Dempster, Manchester. Improvements in connection with apparatus for extracting tar and ammonia from the gases of blast furnaces. July 31.  
10792 J. Farmer, Salford. Improvements in the construction of apparatus employed for bleaching, washing, chloring, scouring, soaping, dunging, and dyeing woven fabrics. Partly communicated by A. Lalancé, Mulhouse, Germany. Complete specification. July 31.  
10811 J. Mactear, Glasgow. Improvements in obtaining iron oxides to be used as pigments. July 31.  
10812 J. Mactear, Glasgow. Improvements in furnaces for chemical processes. July 31.  
10813 J. Mactear, Glasgow. Improved process for obtaining or recovering manganese oxide from manganese sulphate produced in obtaining chlorine. July 31.  
10860 H. E. Newton, London. Improvements in the manufacture of anhydrous sulphuric acid. Communicated by A. Nobel and G. Fehrenbach, Paris. August 1.  
10861 F. Taylor, Wilmington, Delaware, U.S.A. Improved method of treating vulcanized fibre and like material. Complete specification. August 1.  
10869 C. D. Abel, London. Apparatus for distributing liquids in showers. Communicated by La Société Anonyme des Produits Chimiques de la Manufacture de Javel, Paris. August 1.  
10870 C. D. Abel, London. Process and apparatus for purification of alcohol. Communicated by I. A. F. Bang and M. C. A. Ruffin, Paris. August 1.  
10871 C. D. Abel, London. An improved method and apparatus for recovering the nitrous products in the manufacture of sulphuric acid. Communicated by La Société Anonyme de Produits Chimiques de la Manufacture de Javel, Paris. August 1.  
10886 J. Taylor and J. Taylor, Wolverhampton. Improved means for preparing iron and steel sheets and other parts or articles of iron or steel, coating them with copper and alloys of copper. August 2.  
10900 J. G. Willans, West Hampstead. Improvements in obtaining condensable products from coal. August 2.  
10932 H. H. Lake, London. An improved alloy, chiefly designed for electrical conductors, and process of manufacturing the same. Communicated by T. Shaw, Newark, New Jersey. August 3.  
10946 H. J. Haddon, London. A process of coating natural shells or husks with metals. Communicated by H. Peyot, Algiers, Africa. August 5.  
10947 H. J. Worsam, London. An improved apparatus for heating or boiling liquids. August 5.



- 10955 Sir F. J. Bolton, E. J. Hayball, and H. S. Cooper, London. An improved method of producing coloured designs on linoleum, corticine, kamptulicon, and other similar fabrics, and in machinery or apparatus for that purpose. August 5
- 10956 Sir F. J. Bolton, E. J. Hayball, and H. S. Cooper, London. Improvements in printing designs on linoleum, floorcloth, corticine, kamptulicon, and other similar fabrics, and in machinery or apparatus for that purpose. August 5
- 10973 J. Carter, Chelsea. Improvements in filtering and purifying water, sewage, and other liquids; separating the liquids from the solids, correcting the fœtor, and mode of treating or preparing the solids for manure, and other methods of purifying adulterated rivers. August 6
- 10976 R. F. Phillips, Manchester. Improvements in apparatus for broozing, colouring, or otherwise ornamenting and dusting paper or other material. August 6
- 10986 T. Wilkens, Peckham. An improved explosive compound. Communicated by T. Petry and O. Fallenstein, Duren, Germany. August 6
- 10995, T. J. Prout, Plymouth. The prevention of rust on steel or iron in the open air, or in damp places, or near the sea coast. August 6
- 10998 W. F. Shand, London. Improvements in the method of and appliances for the generation of bleaching gas. August 6
- 11002 F. Gaess, Barmen, Prussia. Improvements in the manufacture of red azo-dye-stuffs or colouring matters. August 6
- 11007 F. Brown, London. Improvements in the manufacture of potash salts. Complete specification. August 6
- 11008 F. Brown, London. Improvements in the manufacture of carbonate of soda. Complete specification. August 6
- 11009 F. Brown, London. Improvements in the manufacture of bicarbonate of soda. August 6
- 11023 G. S. Hazlehurst, Rhyl. Improvements in and in apparatus for the manufacture of salt from brine and the concentration of brine. August 7
- 11030 J. H. Johnson, London. Improvements in the preparation of purple colouring matters suitable for dyeing and printing. Communicated by H. Caro, Mannheim, Germany. August 7
- 11035 W. Weldon, Burstow. Improvements in the manufacture of chlorine and hydrochloric acid. Partly communicated by Messieurs A. R. Pechiney et Cie., Salindres, France. August 7
- 11036 W. Weldon, Burstow. Improvements in manufacturing chlorine from hydrochloric acid. Partly communicated by Messrs. A. R. Pechiney et Cie., Salindres, France. August 7
- 11037 F. W. Renault, Burstow. Improvements in indigo vats, and in vats for dyeing with noir bleu or with mixtures of noir bleu and indigo. Communicated by C. Collin and L. Benoist, Paris. August 7
- 11040 A. F. Link, London. Improvements in the construction of coke ovens. Communicated by T. Bauer, Ph.D., Munich, Bavaria. August 7
- 11057 J. Jamieson, Newcastle-on-Tyne. Improvements in the preparation of safety paper affording protection against rasures or other alterations. August 9
- 11097 G. H. Ellis, Exeter. A new or improved process for consolidating granular and other particles or substances into solid crystalline blocks, bricks, stone, and other forms. August 9
- 11104 L. Steinmüller and C. Steinmüller, London. Improvements in the means of and apparatus for extracting oleaginous matters from bones and other greasy substances. Complete specification. August 9
- 11108 F. Bosshardt, London. An improved treatment for the production of raw material for asphalt paving, or similar purposes. Communicated by E. Dietrich, Berlin. Complete specification. August 9
- 11109 W. Bittner, London. Improvements in apparatus for extracting fat, grease, or oil from bones, wool, or other greasy substances. Complete specification. August 9
- 11120 A. O. Walker, Chester. A process for the condensation and deposition of particles of metal and metallic compounds suspended in air or other gases. August 9
- 11127 J. Mactear, Glasgow. Improvements in the manufacture of chemical manures and in apparatus therefor. Complete specification. August 11
- 11151 J. Miller, London. Improvements in the system or appliances employed in the manufacture of bricks, paving blocks, or the like, from the slag or scoria of a blast furnace, the said system or appliances being partially applicable for other purposes. August 11
- 11155 S. P. Wilding, London. An improved mastic or cement. Communicated by J. Aubrey and Count G. de Vanssay, Paris. August 11
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## NOTICES.

In reply to numerous inquiries the Secretary begs to inform Members that the first six numbers of the Society Journal for 1882 have now been reprinted, and can be obtained on application, while the orders already received are being executed with every dispatch. For the information of those desiring to have their copies bound, it may be convenient to know that the index and title page of Vol. I. are to be found in the July number, 1883.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 43, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

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### ERRATUM.

Among the Members elected on the 23rd July the name "Augustus J. Farmer" should read "Augustus J. Fenner."

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CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE,

April 1884.

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## ON THE MANUFACTURE OF GAS FROM OIL.—No. 1. PRELIMINARY NOTE ON CERTAIN BYE-PRODUCTS OF THE MANUFACTURE OF GAS FROM OIL BY THE PINTSCH PROCESS IN RELATION TO THE QUESTION OF THE CONDITIONS UNDER WHICH *BENZENOID*\* HYDROCARBONS ARE FORMED.†

BY HENRY E. ARMSTRONG, PH.D., F.R.S., SEC. C.S.

Professor of Chemistry, City and Guilds Institute, Finsbury Technical College.

In the philosophical transactions for 1825 is a paper "On New Compounds of Carbon and Hydrogen, and on certain other products obtained during the decomposition of Oil by heat," by M. Faraday, which will ever be memorable, one of the *new* hydrocarbons described being *benzene*. In those days the "Portable Gas Company" prepared gas by decomposing oil—I believe a mixture of fish and vegetable oils—at a high temperature, and Faraday tells us that in the operations of this company, when the oil-gas was compressed, a fluid was deposited which could be drawn off and preserved in the liquid state; nearly one gallon of liquid was obtained from 1000ft. of good gas, the pressure applied amounting, he states, to 30 (!) atmospheres.

In recent years, oil-gas has been very successfully employed in lighting railway carriages; and all Londoners must have noticed how brilliant an illumi-

nant it is. A well-arranged plant for its manufacture, storage and application is supplied by Pintsch's Patent Lighting Company, which is in use at the works of the following railway companies:—

	Carriages	
	Fitted.	In hand.
Caledonian .....	112	66
Glasgow and South Western .....	150	50
Great Eastern .....	592	—
Great Western .....	30	8
London and South Western .....	353	50
Metropolitan .....	317	—
Metropolitan District .....	350	—
Midland .....	115	—
North British .....	26	—
South Eastern .....	151	—

The amount of oil-gas made by some of these companies is very considerable. Thus the Great Eastern having 592 carriages provided with the necessary fittings, in the six months January to June of this year, have produced at their works at Stratford 1,302,900 cubic feet of gas, using for this purpose 17,629 gallons of oil. The Metropolitan District have the Pintsch fittings on 350 of their carriages, and during the same period have made nearly 2½ million cubic feet of gas. The London and North Western also make and compress oil-gas, but use a plant arranged by Mr. Pope, which is also in operation at the Great Western works.

During the past 14 years Mr. J. Keith has fitted up in a number of places a patent plant for the manufacture of oil-gas, as a rule for use where coal-gas has not been available. This has been in use for some time past at Langness Point, Isle of Man, to provide gas for a pair of 8-horse Otto engines which operate the fog horn; and at the present moment large works are being erected by the Commissioners of Northern Lighthouses, at Ailsa Craig, Firth of Clyde. The gas will be used there both in the lighthouse and in driving the engines for the fog-horns, but will be reduced before consumption by dilution with air to about 25 candle power, so as to admit of the use of the ordinary forms of burner and gas engine.

Pintsch gas is made by decomposing petroleum or shale oil, and is compressed to about 10 atmospheres; during compression, liquid is deposited in a chamber immediately attached to the pumps, and to a much larger extent in the reservoir in which the gas is stored and from which it is delivered into the iron recipients (drums) attached to the railway carriages; this liquid is commonly called "hydrocarbon," and it will be convenient to speak of "pump hydrocarbon" and "reservoir hydrocarbon." A good deal of "tar" is deposited from the gas prior to its collection in the gasholder.

My attention was first specially directed to the process about *four years ago*\* by my friend Prof. McLeod, and through the courtesy of Lord S. A. Cecil, General Manager of the Metropolitan District Rail-

\* Only the week before I read my paper, I learnt that Mr. Greville Williams had also been engaged in the study of the hydrocarbon, from noticing amongst the list of applications for patents one by him for "A process for the separation of benzene and its homologues from the liquid obtained by compression of petroleum gas." Mr. Greville Williams, in a note in the *Chemical News* of May 2nd, 1881, p. 197,—published in consequence, he states, of his finding that I am working on the subject—gives the following as the results of his examination of seven specimens from stations where the Pintsch plant is in use:—

	Relative density.	Percentage of Benzene and toluene.
A .....	850	65.6
B .....	835	54.2
C .....	840	52.0
D .....	830	45.2
E .....	840	41.4
F .....	800	37.8
G .....	760	24.6

I imagine that these samples were from the seven London companies mentioned in my list; probably it will be found that the difference in composition is mainly the result of difference in the temperature at which the gas is made.

\* This term is used as including all hydrocarbons represented by "closed chain" formulae, such as benzene, naphthalene and anthracene, in contradistinction to *paraffinoid* hydrocarbons, or those represented by "open chain" formulae, such as the paraffins, olefines, acetylenes, etc.

† The substance of this paper was communicated to the meeting on April 7th.

way, I was enabled to procure large quantities of the various by-products for examination, and generally to watch the operations involved. It soon became evident that much information was to be gained by a careful and thorough study of the manufacture, and an approximate examination of the "hydrocarbon" left no room for doubt that I had in my hands practically the same material as that which Faraday had dealt with. Although the investigation is by no means complete, it appears desirable to give a preliminary account of the results, now that the subject of high temperature changes is becoming of such practical importance in connexion with the question of the complete utilisation of coal.

Without entering into details as to the method of examination, which will be described elsewhere, it will be sufficient now to point out that, in the first place, notwithstanding the nature of the material used in making oil-gas, the "hydrocarbon" is practically free from paraffins, containing but traces of hydrocarbons insoluble in sulphuric acid; it essentially consists of benzene and toluene, mixed with hydrocarbons of the  $C_nH_{2n}$  and  $C_nH_{2n-2}$  series. The "reservoir hydrocarbon" especially is saturated with gas, and on passing this into bromine, a solid bromide of the composition  $C_4H_6Br_4$  is obtained, which melts at  $116^\circ C.$ , and is but slightly volatile. The gas is without action on ammoniacal cuprous solution, and is, I believe, identical with the crotonylene (b.p.  $18^\circ C.$ ) separated by Caventou from the hydrocarbons condensed by compression from coal-gas; probably it is *methylallene*,  $CH_3.CH.C.CH_2$ . The "hydrocarbon" also contains normal amylene,  $CH_3.CH.C_3H_7$ , and normal hexylene,  $CH_3.CH.C_4H_9$ , and the hexoylene,  $C_6H_{10}$ , discovered by Schorlemmer, besides others which have yet to be finally identified; the last mentioned hydrocarbon, like crotonylene, is also not a true acetylene, being incapable of forming metallic derivatives. Benzene and amylene appear to be the chief constituents, but the composition of the "hydrocarbon" is subject to considerable variation; I have, however, rarely found less than about 50 per cent. benzene and toluene.\* The "pump hydrocarbon" differs from that deposited in the reservoir only in being richer in the less volatile constituents.

On account of the large proportion of benzene and toluene which it contains, the "hydrocarbon" obtained in the manufacture of oil-gas should be of considerable value; but it does not appear to have attracted in this country the attention it deserves, and is, I believe, all exported to Belgium, where—so rumour says—it is used in varnish making. There is no doubt, however, that the ordinary methods and appliances of the average tar distiller are not sufficiently refined to enable him to deal with this material in a satisfactory manner, and that its exploitation will demand the exercise of some intelligence. The oil-gas manufacturer would also derive considerable benefit if, prior to selling the "hydrocarbon," he were to submit it to a partial preliminary distillation, so as to recover from it the dissolved gas and the hydrocarbons boiling below about  $60^\circ C.$  These constituents being of very high illuminating power, and of no value for any other purpose, ought not, in fact, to be sent off the works, but should be returned to the gas. By heating the residue with slightly diluted sulphuric acid, the paraffinoid hydrocarbons are converted either into viscid polymerides, which are not volatile in steam, or into higher boiling compounds, and after this treatment there is no difficulty in separating the

benzene and toluene by ordinary methods. Benzene of the very first quality may readily be obtained by freezing the fraction distilling near to  $80^\circ C.$

The tar from oil-gas is peculiar, and not only on account of the absence of "basic" and "acid" constituents. Specimens which I examined when the subject first attracted my attention contained practically nothing, which would volatilise in steam, and but the merest traces of naphthalene. It appeared to be all but free from benzenoid hydrocarbons, such as occur in such large quantity in ordinary tar, and to consist almost entirely of undecomposed petroleum hydrocarbons. Specimens from the same works which I recently examined were found to be somewhat richer in volatile hydrocarbons, and contained benzenes and naphthalene in appreciable quantity. The difference may have been due to the employment of higher "heats."

The average tar from another works has given about 20 per cent. of steam distillate. More than half of this, however, was rendered non-volatile by treatment with 4:1 sulphuric acid; the residue was for the most part dissolved by the concentrated acid, and mainly consisted of toluene and other benzenes and naphthalene. In these works it is the custom to pass the gas from the retorts first through two upright iron cylinders or condensers, 8 feet high and 2 feet 4 inches in diameter, where it is cooled, and deposits a *thick tar*, and then to pass it into a kind of scrubber in which it is washed with water: here a *thin tar* is deposited. This thin tar has been found to yield over 50 per cent. of steam distillate, of which about 30 per

#### OIL GAS RETORT—PINTSCH SYSTEM.

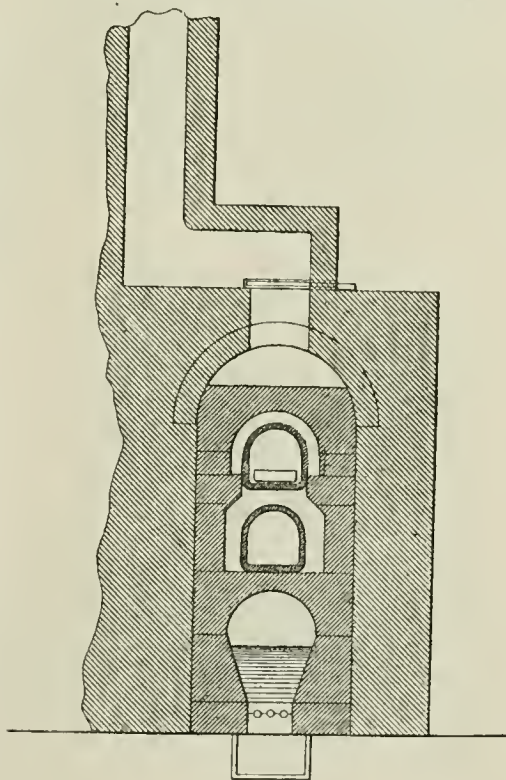


FIG. 1.

cent. is rendered non-volatile by treatment with diluted sulphuric acid; the residue contains but a small amount insoluble in sulphuric acid, and chiefly consists of benzenes and naphthalenes,

\* According to Rudnew (*Dingler* 239, 72), at the Kasan Gas Works, the petroleum gas-tar is distilled, and yields 10-12 per cent. of benzene, about 5 per cent. naphthalene, and some anthracene.



The residue obtained on steam distilling these tars is of greater density than water, and apparently does not contain any of the ordinary coal-tar constituents. The thin tar is of less density than water, and being a saturated solution of the thick tar, very considerable difficulty arises in practice in separating the tar from the water, owing to the tendency to form a kind of emulsion with more or less thin tar on the surface, thick tar at the bottom, and water in between.

In the Pintsch system, two cast-iron D-shaped retorts are set one above the other as shown in Figs. 1, 2, and 3, the largest size in use being 6 feet 2 inches to 6 feet 4 inches long, 10 inches wide, and 9 $\frac{3}{4}$  inches deep; the oil is run into the upper retort at one end, falling upon an iron tray (Fig. 1), which is loosely fitted into the retort, and to complete the decomposition the vapours are passed through the second lower retort. The temperature at which the retorts are worked is very high—a bright cherry red. The oil may be run in at the rate of about 12 $\frac{1}{2}$  gallons per hour. About 80 feet of gas per gallon of oil is reckoned a good yield.

considerable waste of oil in the form of tar. This tar is not so easily disposed of at present, and it must be further studied in order to ascertain its true value; it is not improbable that after separation of the more volatile portion by steam, if not directly available for creosoting, it would be of value for thinning thick creosote oils.

I am indebted to Mr. Rickman, manager of Pintsch's Company for the information that at a recent date the quantities of hydrocarbon and tar obtained per week by several of the Companies were as follows:—

	Hydrocarbon.	Tar, etc.
Caledonian .....	7 $\frac{1}{2}$ gallons.	83 gallons.
Metropolitan .....	91 "	826 "
South Eastern .....	20 "	80 "
London and South Western, 27 "	"	180 "

These figures, together with those already quoted, will give some idea of the extent of production.

The retorts used by Mr. Pope are, I believe, similar to those supplied by the Pintsch Company, but he introduces the oil into the lower retort; probably therefore the higher temperature operates in the

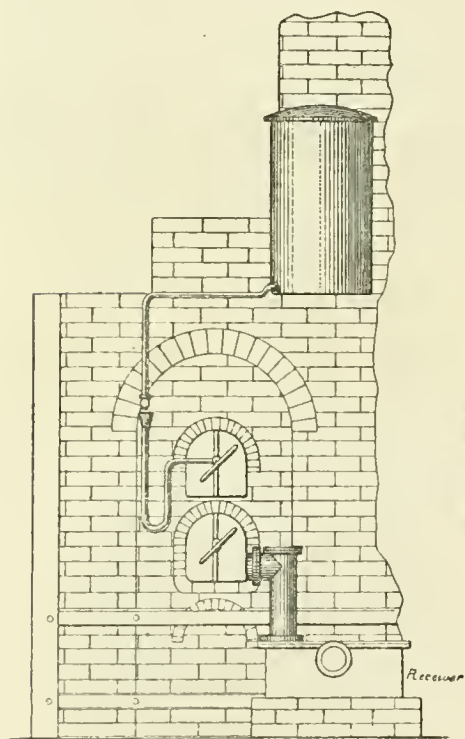


FIG. 2.—SCALE 1 : 30.

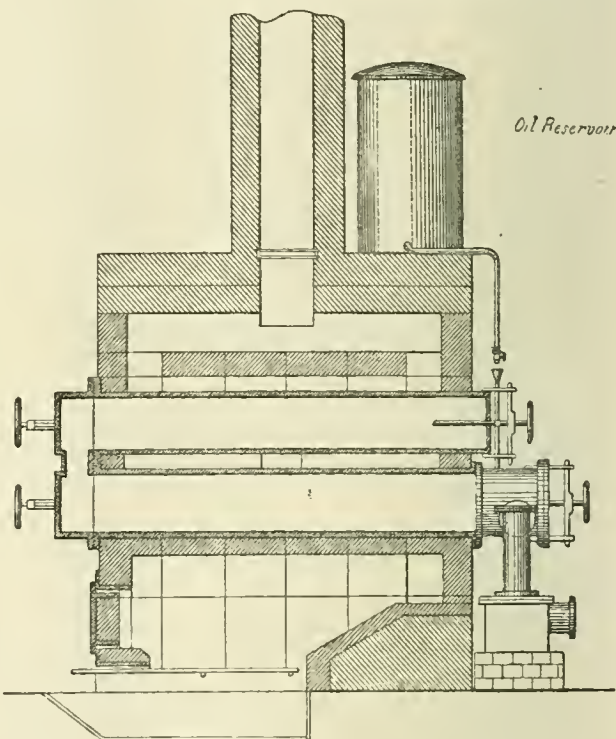


FIG. 3.

So far as I can learn, the quantity of gas produced (and of by-products) does not so much depend upon the quality of oil used as upon the temperature. Somewhat light oils, of about 840 sp. gr., however, it would seem, are now preferred, not so much because they furnish a larger yield of gas, but because they can be worked with greater advantage, causing less furring in the retorts, and with less risk of stoppage of the pipes of the retorts.

The quantity of hydrocarbon deposited may be put at rather less than one gallon per 1000 feet of gas compressed, but the amount depends both on the pressure and temperature (*i.e.* season). Owing to the difficulty of separating the tar from water, the estimates of the amount of tar produced are somewhat unreliable, probably about 5 gallons per 1000 feet of gas made is an average amount, so that there is a

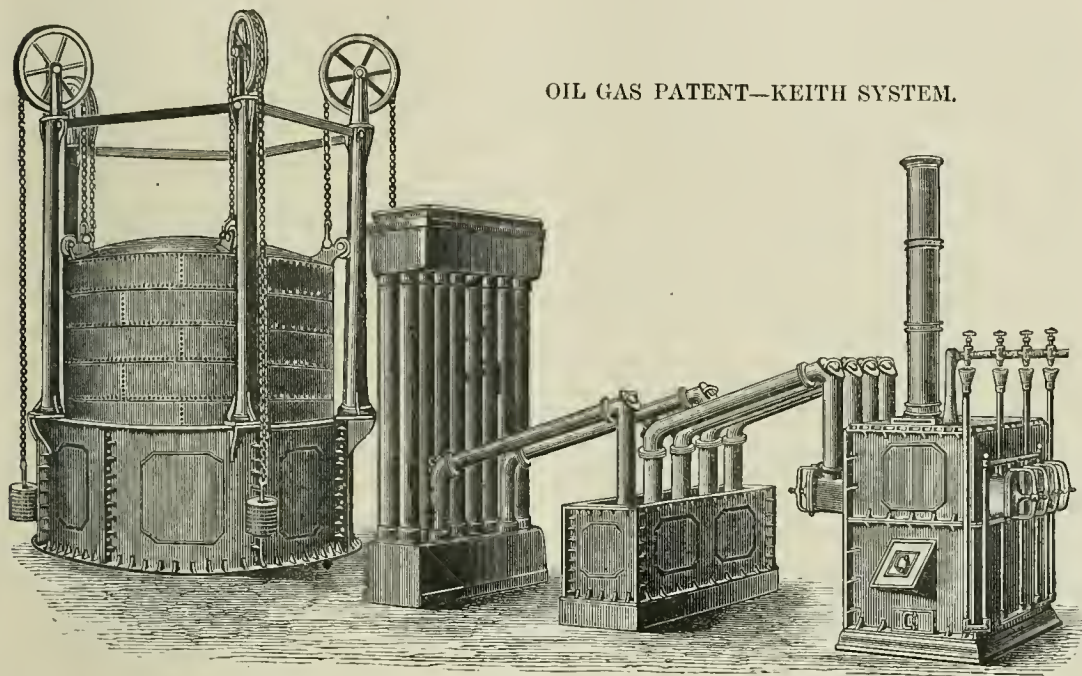
earlier stages of decomposition instead of in the later, as appears to be the case where the Pintsch system is adopted. I have no information as to the results obtained with the Pope plant, and have been unsuccessful in procuring samples of his by-products for examination.

The arrangement of the Keith plant will be obvious on inspection of Figs. 4, 5, and 6. It will be noticed that the retort is of a peculiar form, being constricted in the middle. The largest size of retort, such as will be used at Ailsa Craig, is 6 feet long, 5 inches broad, and 10 inches deep at the end; although shallower in the middle, it is proportionately broader, so that the sectional area is the same as at the end. The oil is caused to flow down an inclined trough not shown in the fig., so that it strikes the retort near the constriction where the temperature is highest.

It is stated that from 100 to 150 feet of gas may be obtained from one gallon of oil, according to the quality of the latter, and with 12 such retorts it is expected that it will be possible to produce at the Ailsa Craig Works at least 2000 feet per hour of not less than 50 candle power.

It is noteworthy that Mr. Keith, and I have this from his own lips, attaches much importance to the quality of the oil, as well as to the manner in which the decomposition is effected.

justifies the conclusion that the character of the hydrocarbons produced depends far less upon the character of the material decomposed than upon the temperature. Thorpe and Young have shown (*Royal Society Proceedings*, 1873, 21, 184) that when solid paraffin is subjected to a moderate heat under a pressure of 20-25lbs., it is resolved into a mixture of lower paraffin and olefines.\* In the oil-gas process, it would seem that a further change takes place which results in the



OIL GAS PATENT—KEITH SYSTEM.

FIG. 4.

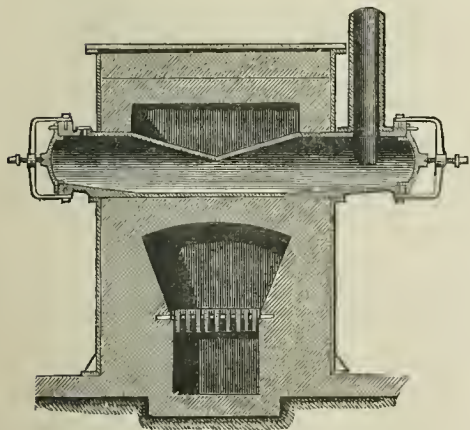


FIG. 5.

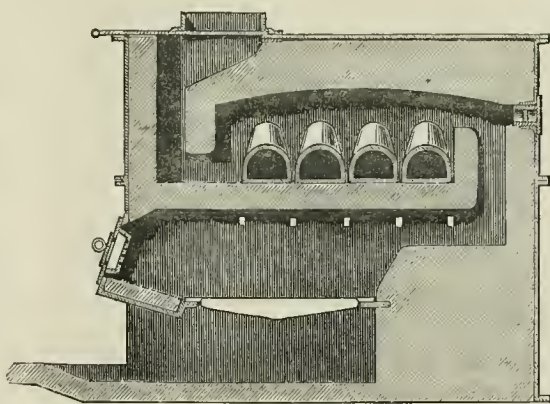


FIG. 6.

On account of the amount of benzene and similar hydrocarbons produced in making oil-gas, it becomes important to consider the bearing of the results disclosed by the study of the manufacture upon the general question of the production of benzenoid hydrocarbons during destructive distillation at high temperatures. Excluding from consideration the phenols and basic compounds which are contained in ordinary tars, the evidence at our disposal, I think,

almost complete destruction of the paraffins and in the formation of benzenes and naphthalene as well as of hydrocarbons of the acetylene series.

The question then arises—How are the benzenoid

\* It is not improbable that hydrocarbons of the acetylene series were also formed; their presence might serve to explain Thorpe and Young's observation that the proportion of paraffin to olefine becomes gradually larger as the molecular weight increases.



hydrocarbons formed? are they derived from corresponding paraffins which have lost hydrogen and, as a more or less direct consequence, have become "closed chain" compounds, or are they built up from simpler materials? and what are the conditions which favour the production of different hydrocarbons? These questions are not only of theoretical interest; it is clearly of importance that we should answer them in order that we may determine what are to be regarded as theoretical conditions and theoretical yields, otherwise the practical man will be without guidance. It has long been held that benzene is formed by the coalescence of three acetylene molecules, and Jacobsen has put forward the hypothesis that toluene is formed from two acetylene and one methylacetylene molecule, the xylenes from one acetylene and two methylacetylene molecules, and mesitylene and pseudocumene from three methylacetylene molecules; but although the formation of all three xylenes may be accounted for, only two of the trimethylbenzenes can be thus produced, but they are the two which alone are present in coal-tar, so that the facts appear to support this hypothesis. It is also well known that Berthelot has synthesised benzene from acetylene. Professor Thorpe informs me that a series of experiments which he has made with Mr. Septimus Dyson confirm Berthelot's statement, but that the amount produced is extremely small; he adds that a comparatively low temperature is needed, much lower, in fact, than he had been led to believe, and that their first trials gave no results from employing too great a heat.

I have been led to doubt the correctness of the popular explanation of the formation of benzene by the observation that not only the liquid deposited on compressing oil-gas, but also the gas itself, is without action upon ammoniacal cuprous solutions, which involves the somewhat remarkable conclusion that acetylene and true homologues of acetylene are absent from the gas. It is noteworthy, however, that in a paper read before the Berlin Chemical Society in 1868 (*Berichte* 1, 88), describing Hirzel's apparatus for the production of gas from petroleum, Dr. Martius states that the gas is rich in acetylene.

A number of observations have been made within recent years which also tend to throw light on the question raised above and are highly instructive. Thus Atterberg (*Berichte*, 1878, p. 1222) passed Swedish wood-tar through iron tubes filled with coke, heated either to dull or to bright redness. The product

obtained at a bright red heat contained all the essential constituents of coal tar: it was rich in naphthalene, contained a fair amount of anthracene, but only small quantities of phenols, and about 7 per cent. of benzene and toluene. At a dull red heat, 50 to 60 per cent. of the tar escaped unchanged; naphthalene could not be discovered in the product, and only 0.3 per cent. of anthracene was detected by the chromic acid method; 10 per cent. of toluene containing but little benzene was obtained, and higher phenols with but little phenol itself. Letny (*Berichte*, 1878, p. 1210), whose results were published in Russian in 1877, made experiments on a large scale in a retort 7 ft. long and 1 ft. across, with Baku petroleum residues (sp. gr. 870; b.p. +270°C.) which were led over red hot charcoal. Tar equal to about 40 per cent. of the petroleum was obtained of the following composition (sp. gr. 1207):—

Below 200°C.	13.9 per cent.	{ 2.3 per cent. water.
	1.6 "	{ benzene below 90°.
	5.2 "	{ toluene, &c., 90°-115°.

From 200° to 270°	26.9 per cent.	{ naphthalene and
From 270° to 310°	8.6 "	{ unchanged petroleum.

The portion boiling above 340° was collected in three fractions.

(a) 12 per cent.	{ 9.3 per cent. petroleum.
	{ 2.7 " crude phenanthrene
	= 76 per cent. pure do.
(b) 7.5 "	{ 5.2 " petroleum.
	{ 2.4 " crude anthracene,
	= 8 per cent. pure do.
(c) 8 "	{ 6.04 " petroleum.
	{ 1.94 " crude anthracene,
	= 1.5 pure do.

Besides tar, a mobile liquid was obtained consisting of benzene, toluene, xylene, and naphthalene, almost free from bodies of higher boiling point.

Two experiments are quoted with tubes 4 ft. long, the one being two and the other but one inch in diameter. Using the former, 100 grams petroleum gave 23.9 grams tar (sp. gr. 880) and 30.1 litres of gas which burnt with a smoky flame; whereas using the latter, 46.4 grams of tar (sp. gr. 890) and 59 litres of gas were obtained. It was observed that when the tube was not packed with charcoal, there was a tendency for it to become stopped by deposition of carbon, and that as soon as it became coated with carbon the decomposition took place with greater regularity. When the tube was packed with platinised carbon, 100 grams petroleum gave 66.6 grams of tar (sp. gr. 890) and 75.2 litres of gas which burnt with a smokeless flame. In experiments on a small scale, using narrow tubes, no solid hydrocarbons were obtained, but only benzene, toluene, xylene, and (?) cumene, mixed with amylene, hexylene, and heptylene in relatively small quantities. Letny therefore attaches great importance to the presence of porous carbon, and states that the quantity of solid hydrocarbons produced is greater in proportion as the layer of carbon through which the vapours must pass is thicker.

Liebermann and Burg (*Berichte*, 1878, p. 723) studied the effect of passing lignite-tar oil, coal-tar, and petroleum through red-hot tubes one metre long and three centimetres wide (brass gas tube) packed with charcoal, or coke, or pumice, in pieces the size of hazel nuts. Lignite-tar oil (sp. gr. 915-920 and 889; b.p. +190° and +230° C.) which is akin to shale oil, was converted into a tar similar to ordinary coal-tar. The amount of gas produced was greatest when the tube was empty, and greater when it was packed with coke or pumice than with charcoal. A large quantity of gas was obtained from commercial petroleum, and a not inconsiderable amount of benzene, but scarcely any anthracene.

Salzmann and Wichelhaus (*Berichte*, 1878, p. 1431), have also made experiments with lignite-tar oil (sp. gr. 871, b.p. +250° C.) using a narrow iron tube packed with the following materials: (1) fibrous asbestos platinised; (2) wood charcoal in pieces the size of peas; (3) bone charcoal in pieces twice as large; (4) pumice in pieces of the same size as the bone charcoal; (5) bone charcoal in pieces the size of lentils. Their results furnish further proof of the influences of "contact substances," but they are not stated in such a form as to give much information or to be directly available for the present discussion.

Taking these various observations into account, especially Letny's important experiments on a large scale with a retort charged with charcoal, and bearing in mind the results of my examination of the products of the ordinary process of oil-gas manufacture, in which an "empty" retort is used, there can be no doubt that while a high temperature is necessary for the formation both of benzene and anthracene, the latter is only produced under special conditions; indeed it would almost appear that its production is dependent on the presence of heated carbon. Obviously, however, very much remains to be learnt before it will be possible to say what are the precise conditions to be observed if it be desired to produce the one or the other.

It is not a little remarkable that an industry of such magnitude and importance should so long have been without the chemist's guidance, especially as the progress of chemistry has received such an impetus from the study of coal-tar constituents. The distillation of coal has been left to the engineer: attention has been almost solely directed to the production of gas. Now that other products than gas are claiming attention, it is high time that the problem should be studied in a systematic and scientific manner, and that the chemist, not the mere analyst, should find a place in the coke and gas works.

The recent attempts to recover much that has hitherto been lost in coking coal are of the greatest importance, and have already led to results of high interest. In a recent paper in our Journal, Mr. Watson Smith has given an account of his preliminary examination of the tars from the Jameson and Simon-Carvès ovens, and has pointed out that the former contains but small quantities of the characteristic constituents of coal tar. I can endorse these statements, having had an opportunity, thanks to Mr. Jameson, of examining his oil. Evidently the Jameson oven gives a product which closely resembles shale oil in composition, and hence is at present of comparatively little value; in comparison with the Simon-Carvès oven, however, it has the great practical merit of cheapness. I have recently had the opportunity of seeing the two systems in operation, and am satisfied that the proposed adaptation of the ordinary beehive oven has not yet received the attention it deserves, and that the arrangements which have been made for condensing and collecting the products are insufficient.

The time must come when coal-tar will not be regarded as a mere bye-product, and when attention will be paid not only to the manufacture of gas, but also of coke and of *particular constituents* of coal-tar: when, in short, the materials latent in coal will be progressively utilised. It may be that then the coal will first be coked, the oil which distils over being carefully condensed, and that the "weak gas" thereby produced will be utilised as fuel; at the same time the ammonia and sulphur will be recovered. The paraffin and whatever else of immediate value it may contain having been separated from the oil, the residue will be utilised in the production of oil gas and of benzene, anthracene, &c. Gas is daily becoming a more important article of consumption, and we may look forward to its use by the public in the near future, not only as an illuminant and in gas engines, but also as a heating agent. The late Sir C. W. Siemens powerfully advocated a double supply, one of heating and one of illuminating gas, of which the former would necessarily be inferior, the latter superior in quality to the present supply if the system he advocated were adopted of collecting in separate holders the gas given off at various periods during the distillation of coal; and this would also be the case if water gas came to be employed for heating. Such a system would involve a double set of mains, and that for the heating gas would have to be of enormous size if gas were to be generally used as a heating agent. The proposal to introduce a gas rich in carbonic oxide for general household use, which the supply of water-gas would involve, might also meet with considerable opposition, and it must be admitted that the mass of evidence on this subject recently collected in the enquiry at Massachusetts is not altogether reassuring. Lastly, I am of opinion that a gas of lower heating power than that at present supplied will be of little use to the general public as distinct from manufacturers; it is impossible with the stoves hitherto contrived and with the present gas supply to raise a surface to such a temperature that it will

radiate sufficient heat to render it possible to use gas with economy in heating rooms in the manner which probably will always be preferred in this country, and therefore I venture to think that it will be better to have but a single set of mains and to supply a gas of much higher illuminating power, and consequently of higher heating power than that we now have. By combining the present system with the manufacture of oil-gas and systematically separating certain of the liquid by-products and re-incorporating them with the gas, this could readily be done, and the oil required might well be that obtained in coking coal.\*

Mr. Pettigrew, at my request, has had the kindness to convert a quantity of "Jameson oil" (sp. gr. 924) into gas; he obtained 71 feet per gallon—a rather low yield, and as was anticipated, the tar produced was very thick and such as would readily clog the pipes. Difficulties of this kind, however, would probably soon disappear in practice.

The problem I have sketched out is a very important one for this country to solve, and its solution would well repay a large expenditure; it is, I fear, too much to hope that those interested will be prepared to incur the outlay required for a systematic series of experiments under the direction, say of a committee of experts constituted of representatives of all the various industries concerned.

\* The Americans have already much experience in the use of such gas, so-called water-gas—really a mixture of oil and water gases—being supplied by over 100 companies in the United States. Much information on this subject may be gathered from the "Report of the Commissioner on Health on Illuminating Gas made to the Committee of Lamps and Gas of the Common Council of the City of Brooklyn, July 3rd, 1883," and from the Report of the hearing, during this year, before the Joint Standing Committee on Manufactures of Massachusetts, of a petition to amend statutes relating to manufacture of illuminating gas. Nearly one-half of New York city is stated to be now supplied with gas made by the Tessie-du-Motay process. The Municipal Company manufactured last year 913 million feet of 29 candle power, and the increase of sales was about 15 per cent, during the year, which, as Mr. Morse in his argument for the appellants in the above hearing points out, is an indication of the estimation in which "water-gas" is held by the public, since there is an agreement between the coal gas and water gas companies in New York city not to interfere with each other's business, and their business increases only as the customers of one company insist on going to another. He says also that the water-gas there is daily making inroads upon the coal gas, for the reason that the gas is cheaper, heavier, has more candle power and less smoke(!), and is much more brilliant than coal gas.

In a paper on the Illuminating Gas of New York City, by Dr. Love, gas examiner, read at the meeting of the American Association for the Advancement of Science, at Boston, in August, 1880, and reprinted in the above-mentioned Brooklyn Report, a number of interesting facts are stated, relating to the manner in which the New York Companies manufacture their gas. The Metropolitan Company, he says, calculate upon getting 70 feet of 60 candle gas per gallon of oil. The following table may be quoted, giving his results of analyses of determinations of average illuminating power:—

	Manhattan Co.	Hudson Co.	Metropolitan Co.	New York Mutual Co.	New York Co.	Municipal Co.
Hydrogen .....	45.79	46.53	35.41	10.57	27.14	26.25
Methane .....	39.01	12.38	42.66	41.75	25.35	28.91
Carbon monoxide .....	6.31	3.11	9.17	9.53	26.81	27.12
Carbon dioxide .....	—	1.08	—	1.51	3.02	—
Nitrogen .....	2.51	0.50	5.35	20.69	2.87	1.92
Oxygen .....	—	0.06	—	0.54	0.15	—
Illuminants .....	6.38	6.31	7.41	15.11	14.63	15.80
Candle power .....	—	—	20.33	26.53	21.35	29.68

The large proportion of nitrogen in the Mutual gas will attract attention: I do not know whence it is derived. This company manufacture the body of their gas partly from wood and partly from coal, of which the distillation is pushed to a yield of 15,000 feet per ton, and enrich with oil gas. The proportion of carbon monoxide is low when it is remembered that after each charge of wood is distilled, the charcoal is pushed back into the boat (the retorts are of a peculiar shape), forming a solid mass of heated charcoal, through which all the heated gas has to pass before reaching the stand pipe.



I cannot better conclude this note than by acknowledging my indebtedness to Mr. E. de M. Malan and Mr. W. F. Pettigrew, engineers in charge of the oil-gas works of the Metropolitan District and Great Eastern Railway Companies respectively. Having received permission to assist me in this enquiry these gentlemen have done their best in every way to aid me.

In a future paper I propose to consider the composition of oil-gas and the temperature at which decomposition is effected.

#### DISCUSSION.

The CHAIRMAN said: The subject which Dr. Armstrong has brought before us is specially interesting not only as regards the valuable Pintsch process but as regards the wider question of the conditions under which hydrocarbons of the benzene and paraffin series are respectively produced in destructive distillation, the former of which are so much more valuable than the latter.

Dr. PERCY F. FRANKLAND said: He should like to ask Dr. Armstrong whether he had analysed the gas produced in Pintsch's process, and whether he had found that as poor in hydrocarbons of the paraffin series as was the tar. Some years ago he had occasion to make an elaborate examination of a patent process for the manufacture of oil-gas; the tar produced in that process he had not examined, as he was only interested in the quality of the gas itself. This gas he found to contain a very considerable proportion of paraffin-hydrocarbons, speaking from memory they amounted to 12 to 20 per cent. The presence of these paraffins in the gas, although apparently contrary to Dr. Armstrong's experiences with the tar, was probably owing to the temperature of distillation having been considerably lower; in his experiments the retorts were only kept at a cherry-red heat, whilst in Dr. Armstrong's the temperature appears to have been bright orange. Probably at the high temperature thus used in the Pintsch process these gaseous paraffins were converted into hydrocarbons of the benzene series.

Mr. W. F. PETTIGREW said: He was sorry he could not follow the gentlemen who had spoken of the by-products (hydrocarbon and tar) obtained from the manufacture of oil-gas in a scientific manner as a chemist, but as one who had charge of works where a large quantity of oil-gas was made he could say a few words as to how the hydrocarbon and tar were obtained. As Dr. Armstrong had said, for every 1,000 cubic feet of gas made and compressed, about one gallon of liquid hydrocarbon is obtained, besides a quantity of tar, the 1,000 cubic feet of gas being made from about 12½ gallons of oil (the oil being about 840 gravity and flashing at about 180° to 200° F., open test.) When the works were first started, the hydrocarbon and tar were thrown away into the ditches and drains round the works as useless, although sometimes a little of the tar was used for tarring the timber, but was objected to as it would not dry and had a disagreeable odour. As complaints were made of this causing a nuisance and obstruction, endeavours were made to find a market, and after some time 6d. a gallon was obtained, then 9d. and at the present time the price is about 1s. 5d. to 1s. 6d. a gallon. The use and destination of the hydrocarbon does not seem known except that it must be of greater value in Belgium, where it was probably to be sent, the price paid there including cartage and carriage by vessel and rail. The tar is probably also sent elsewhere in the continent, as it is loaded in barges and sent to the docks for shipment in deliveries of about 50 casks each time. The price of the tar varies ½d. to 1½d. per gallon.

Mr. G. C. TREWBY said that it was an error to suppose that in the manufacture of coal-gas by the London companies, naphthalene was deposited from the gas in any considerable quantities; that it was only in the service pipes to consumers in the district where the pipe was exposed that naphthalene was crystallised out of the gas, and then, in clearing the obstruction, it was blown back into the main and taken up again by the gas; that no appearance of naphthalene was found in the large mains, and the quantity deposited in the works was infinitesimal. In the tar as made from coal supplied from the Durham district, it was well known that naphthalene existed largely but in the tar produced from the distillation of the Midland and cannel coals very little naphthalene was found. In the case of a deposit of naphthalene in the service pipes owing to a change of temperature, it could be got rid of by increasing the illuminating power of the gas one or two candles by the use of cannel, the richer gas acting as an absorbent and keeping the naphthalene in suspension, but, of course, the gas companies having to supply gas of a fixed standard of illuminating power, did not get paid for the extra quality which they gave under these circumstances.

Mr. H. W. LANGBECK said: One of the residues obtained in distilling shales is the so-called black oil; if light falls upon it, it appears dark green, if light passes through a thin stratum, reddish brown. Its specific gravity is 0.880 to 0.885; it boils between 150 to 200° Cels. This oil is used for rendering leather waterproof, keeping it supple at the same time, the only objection to using it thus is its disagreeable smell, which cannot be got rid of. Lately, the residue black oil obtained from petroleum has been imported from the United States for the same purpose. It is so far preferable, as it is nearly devoid of smell.

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## Journal and Patent Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

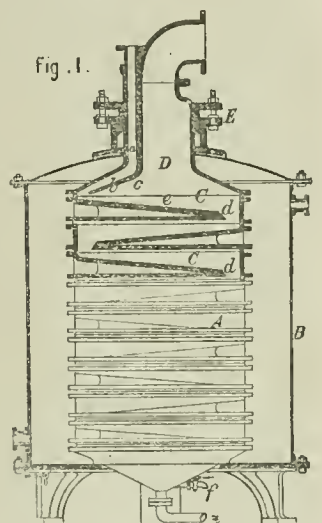
*Danger to Steam Boilers from Ferric Sulphate.* K. List. *Chemisches Centralblatt* 15, 351.

A YELLOW deposit, consisting chiefly of ferric sulphate, is frequently observed to occur on the external surface of the boiler. In consequence of a boiler explosion, the author made some investigations upon it. Such deposits consisted of a basic compound occasionally of the composition  $5\text{Fe}_2\text{O}_3 \cdot \text{SO}_3$ ; they dissolve slowly in a little water, but on heating with much water a reddish yellow precipitate, rich in ferric oxide, results, free sulphuric acid or an acid salt going into solution, which now dissolves metallic iron with evolution of hydrogen, giving occasion for the formation of fresh basic iron salts. As this process is repeated, so will the boiler be more and more attacked. Such ferric sulphate can be formed on the exterior of the boiler by the action of the products

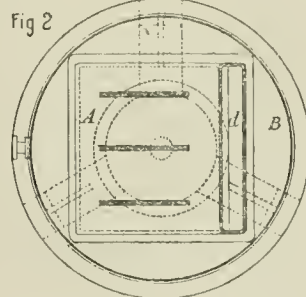
of combustion of coal containing sulphur, especially with the aid of steam; but in the interior of a steam boiler, free sulphuric acid or ferric sulphate, even in small quantity, must act injuriously, as has been actually proved in the case of a perfectly new boiler. Care should therefore be taken that both should be avoided as much as possible. Owners of boilers should avoid bringing into contact with their boilers water which contains free sulphuric acid, however little, and should prevent the formation of ferric sulphate on their boilers. If a boiler water shows an acid reaction, it is most readily neutralised by a small quantity of soda.—F. L. T.

*Apparatus for the Continuous Evaporation of Liquids.* H. Egells, Berlin. Ger. Pat. 27162, 1883. *Dingl. Polyt. Journ.* 253 [4] 155.

THE apparatus consists of the steam-jacket B (Figs. 1 and 2) and the concentrating vessel A, which is provided alternately on both sides with pockets C, open to the outside. Along the side of the dome D is the feed-pipe *a*, from which the liquor to be concentrated runs into a groove *c*, spreading over the whole width of A.



From *c* the liquor proceeds to a distributing plate C, and thence in a wide thin layer to the first pocket C, which is slightly sloping at the top, but makes a sharp angle at the end *d*; the bottom is horizontal. The liquor travels only slowly on the top of the pockets until it



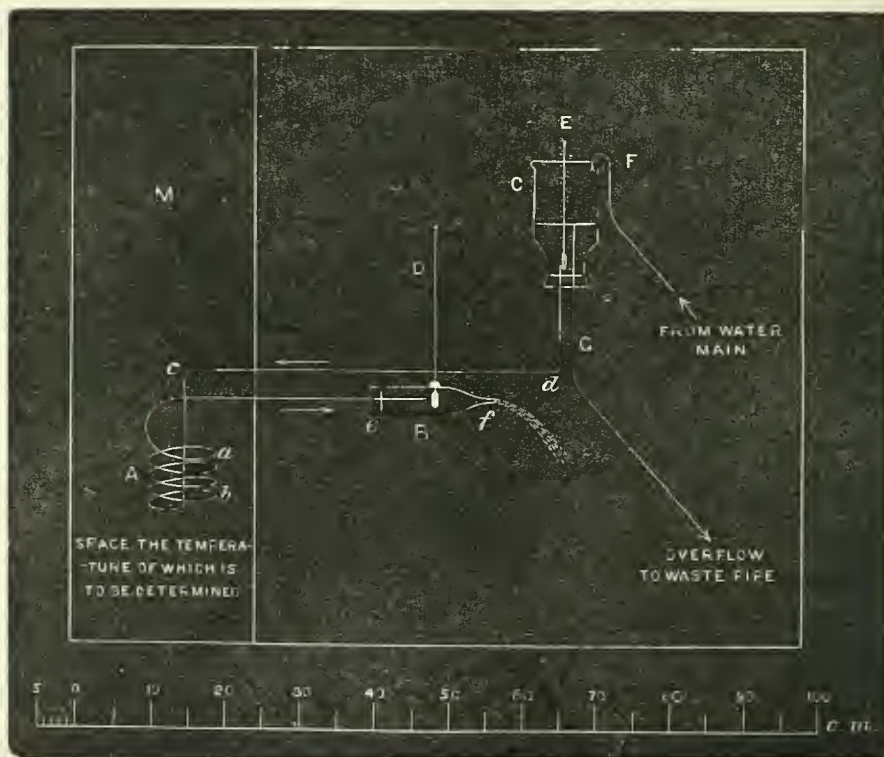
arrives at *d*, where it is torn off, as it were, and drops in distinct thin streams on to the next pocket. At the bottom there is a tap *f*, to draw samples. If acids are to be concentrated, the inside cylinder is made of lead, porcelain or stoneware, whereas the outside jacket can be made of metal in all cases. To allow for the difference of expansion of the inside and outside cylinder, the dome D passes through a stuffing box E, above which it is connected with an elbow-pipe, and so forth, to a still or a receiver, or a condenser and air pump.—S. H.



## II.—FUEL, GAS, AND LIGHT.

*A New Form of Pyrometer.* Thos. Carnelly, D.Sc., and Thos. Burton. Chem. Soc. Journ., July, 1884.

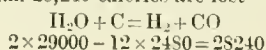
THE principle of the new apparatus is as follows:—"If a current of water of known temperature be allowed to flow at a constant rate through a coiled metallic tube placed in the space; the temperature of which is required, then the increase in the temperature of the outflowing water will be the greater the higher the temperature of the space." The arrangement will be readily understood from the following sketch, which is drawn the actual size of the apparatus used by the authors.



A is a coil of copper tubing placed in the space M, of which the temperature is required. A current of water flows from the cistern C, through the coil A, into the glass tube B, and out at the nozzle f. The tube F, attached to the water main, supplies the cistern at such a rate as to maintain a constant overflow through G; a uniform head of water is thus obtained. The effluent branch of the copper coil projects such a distance into the tube B that the issuing water flows directly on to the bulb of the thermometer D. The copper tube has an internal diameter of 6mm. It is necessary to graduate each instrument by exposing it to known temperatures, and to construct a table from the data obtained. Each variation in the flow of water will of course necessitate a fresh graduation. The authors do not claim great accuracy for their instrument, but consider it to be sufficiently reliable for manufacturing operations. A similar instrument has been recently devised by Messrs. Boulier Bros., and described by Louth (Bull. Soc. Chim. 40, 105).—E. J. B.

*Application of Steam in Generators.* By R. Schöffel. Chemisches Centralblatt 15, 414 and 431.

THE author was so surprised at Schmidt's results (Chem. Centr. p. 203) that he felt obliged to control the calculations. Schmidt calculates that in the gasification of 18 parts of steam 28,240 calories are lost—



these he replaces by the gasification by air of a further amount of carbon =  $\frac{28240}{2480} = 11.38$ , and concludes that for the gasification of 12 parts carbon by 18 parts steam, 11.38 parts carbon require to be gasified by air, or that 77.9 parts of steam can be used to 100 parts carbon. The author points out that Schmidt calculates merely sufficient carbon to make the evolution and absorption of heat equal, so that the ultimate evolution of heat in the generator would be nil. The temperature of a generator containing pure carbon driven gently by air is, by calculation,— $T = \frac{2480}{2.333 \times 0.24 + 4.463 \times 0.24} = 1520^\circ$ . It is

to be observed that such a high mean temperature in the generator is not necessary, otherwise the injection of the smallest quantities of steam would theoretically be *a priori* inadmissible. According to Unger (Liebig's Ann. 63, 240), the reduction of carbon dioxide by carbon does not take place at 1000°, nevertheless at 1300° the carbon dioxide begins to dissociate, so that 1200° may be taken as the minimum temperature at which the generator can be worked. On this supposition the amount of carbon can easily be calculated—

$$1200 = \frac{12 \times 2480 - 2 \times 29000 + 2480x}{2.333(12+x)0.24 + 2 \times 3.4 + 4.463x \times 0.24}$$

whence  $x=85$ . Accordingly 85 parts must be gasified by air when 12 parts are gasified by steam, and to 100 parts of carbon may be allowed at most 18.5 parts steam, instead of 77.9, according to Schmidt. The composition of the generator gases will then be—

Carbon monoxide	$(85+12)2.333=226.3$	or 37.25 per cent.
Hydrogen	$= 2$	0.33 "
Nitrogen	$85 \times 4.463 = 379.3$	62.42 "

607.6

whilst the composition of the gases when working with air alone would be—

Carbon monoxide	$(85+12)2.333=226.3$	or 34.33 per cent.
Nitrogen	$(85+12)4.463=432.9$	65.67 "

639.2

If the amounts of heat yielded by an equal weight of carbon in the gases are calculated in the two cases, it shows—

In the 1st case  $226 \cdot 3 \times 2400 + 2 \times 20000 = 601,120$  calories

In the 2nd case  $226 \cdot 3 \times 2400 = 543,120$  „

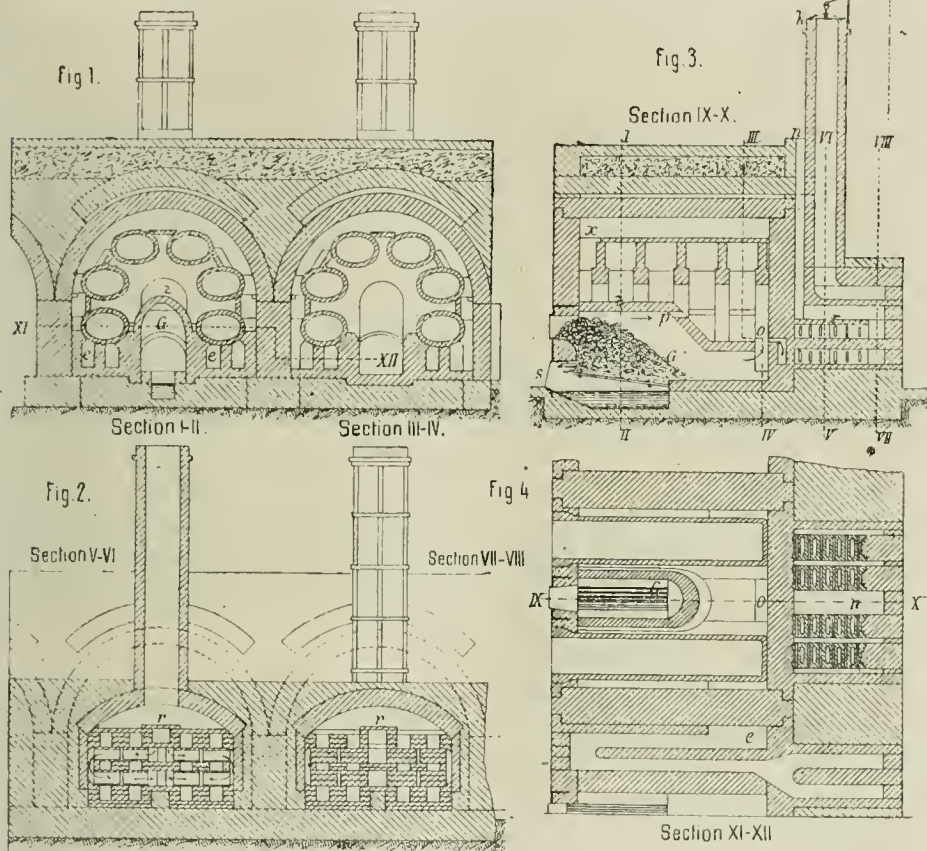
Difference - - - 58,000 „

This difference turned into carbon =  $\frac{58000}{8080} = 7 \cdot 17$ . As

*Preparation of Illuminating Gas.* A. Klönne, Dortmund. Ding. Polyt. Journ. 253 [5], 202. Ger. Pat. 25157, 1883.

THE producer G (Figs. 1 to 4) is separated from the retorts by an arch *z*. The producer-gas enters through the openings *o* and *p*. At *o* it comes in contact with the hot air from the regenerative furnace, and is burnt. The fire-gases then proceed to the front to *x*, and travel again to the back to the flue *e*, where they heat on their

### A Klönne's Regenerative-Furnace for Gas-Retorts. (1-10)



the amount of carbon used is 97, this shows an apparent saving of 6·9 per cent. It is obvious that the difference in the two amounts of heat is due to the two units of hydrogen, the heating effect of which was consumed in the generator, and reappears in the gases. By the reduction of the temperature of the generator from 1520° to 1200°, an amount of heat equal to 58,000 calories has been stored in the combustible gases in the form of hydrogen. The gases generated at 1200°, with the help of steam, contain an amount of heat

$$605 \cdot 6 \times 1200 \times 0 \cdot 24 + 2 \times 1200 \times 3 \cdot 4 = 182573$$

those prepared at 1520 by air alone

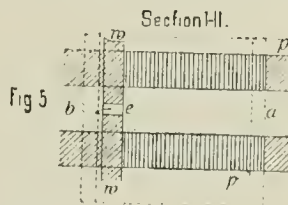
$$659 \cdot 2 \times 1520 \times 0 \cdot 24 = 240476$$

Difference - 57903

If it were possible to utilise the gases with their full initial temperature, they would both have the same effect. It is only when the gases have to be conducted some distance, so that they are cooled before use, that there is any advantage in the employment of steam, and even then only within the calculated minimum limit.

—F. L. T.

way the bottom of the lowest retort. They then pass through the regenerator *r* and escape to the chimney. The air necessary for combustion enters at *n*, travels in circular flues round the regenerator, and, as mentioned above, is mixed at *o* with the producer-gas. The formation of CO is regulated by the door *s* and the damper *K*. To increase the strength and working power of furnaces



for gas retorts, the inventor (Ger. Pat. 26906, 1882) uses a layer of firebrick slabs (Figs. 5 to 10), placed at the top of the retorts. He thus avoids loss of heat by radiation, and protects the arch from the direct flame. The pillars *p* are provided with channels, where they touch the retorts, and by this contrivance a large number of pillars



can be used without diminishing the heating surface. The walls *w* divide the furnace in compartments *a* and *b*, and cause the combustion gases to rise first straight

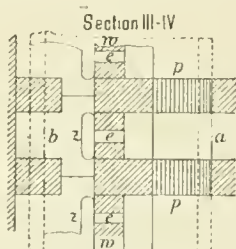


Fig 6

upwards, and afterwards drop down vertically at *b*, whereby all retorts are uniformly heated. In the com-

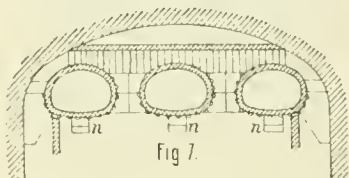


Fig 7.

partment *b* the heating of the bottom of the lowest retort is effected by applying slabs *d* (Fig. 9), furnished

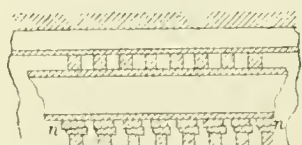


Fig 8.

with slits *z*, in order to compel the combustion gases to travel close to the retort in the direction of the arrow in

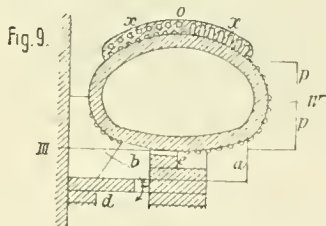


Fig 9.

the figure. This layer of slabs also separates the furnace proper from the regenerator. The retorts rest on pillars provided with wedges *n* (Figs. 7 and 8), which can be

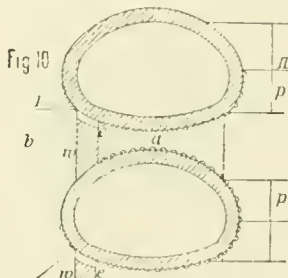


Fig 10

easily moved. To avoid the heating of the retort from the top, which would cause a decomposition of the coal-gas, the upper half of the retort is covered with firebrick slabs *o* provided with holes *x*.—S. II.

*Improvements in or Relating to Hydrocarbon Furnaces.* Eng. Pat. 5357. Nov. 13, 1883. A. J. Boulton, London. A communication from O. D. Orvis, of Chicago.

THIS is an improved method for supplying liquid hydrocarbons in a vaporised form to fire chambers, whether these latter contain a bed of solid carbonaceous fuel or not. An inlet pipe passing through the front wall of the fire chamber has upon its outer and projecting end a globe vacuum chamber. Into this globe pass an air-pipe and a steam jet—this latter reaching beyond the centre of the globe, and opening in a line with the inlet pipe. Between the globe and the furnace wall is an oil nozzle, projecting downwards into the inlet pipe, and so delivering that the oil escaping from it will be in the same line with the steam issuing from the jet, and as near to this latter as practicable. The steam may be superheated by passing the pipe bringing it from the boiler through the furnace fire. By these means proper proportions of steam, oil and air can be admitted to the furnace, but as with this construction only the lighter portions of the hydrocarbon would be vaporised, and the heavier portions thrown into the fire chamber in a liquid form, a further part of the invention provides for so perfect a vaporisation that a jet of mingled steam, hydrocarbon vapour, and air is projected into the furnace in a sheet-like form, well adapted for quick and thorough ignition. With this object, upon the inner end of the inlet pipe is screwed a retort constructed of such material as to withstand the heat of the fire to which it is exposed. The bottom of this retort falls below the level of the inlet pipe, so that it forms a receptacle for the liquid portions of the hydrocarbon blown forward by the steam jet. At the top of the retort, and opposite to but above the plane of the inlet pipe, is a horizontal slot through which the combined blast of steam, hydrocarbon, and air enters the furnace. It will be seen that the heat of the fire vapourises the contents of the retort, and they escape under pressure in the form of a sheet by the slot above mentioned. Where it is not convenient to use the retort, a nozzle embodying the spirit of the invention may be fitted to the inlet pipe instead.—A. R. D.

*Improvements in the Manufacture of Coke for Obtaining Products therefrom, etc.* Eng. Pat. 5577. Nov. 29, 1883. A. B. Cowan, Spennymoor, Co. Durham.

THE gases passing from the scrubber in a process like the Jameson still contain considerable quantities of oils, to recover which is the object of the present invention. After passing through the scrubbers the gases are conducted through a pipe diminishing at one point, the contraction being, as it were, formed by the meeting of the small ends of two truncated cones. At the smallest part of the pipe an annular steam jet is introduced, which produces in the gas passing through the contracted orifice a much greater velocity than in any other part of the pipe, and develops in the oil a tendency to separate from the gas, the impetus of the particles of the former being greater than the impetus of the particles of the latter. It is preferable to add after this arrangement a further scrubber with loose packing for the more effectual removal of the oil. The steam jet may be made of such size and power as to allow other exhausting apparatus to be dispensed with. Another part of the invention refers to the automatic separation of the oils and liquor condensed in the process. These are led into a tank provided with two outlets on the side opposite to the inlet, and also with baffle walls to cause the contents of the tank to take a lengthy course from their entrance to their exit. Both the outlet pipes deliver at the height to which the tank is intended to be filled; one opens directly from the tank at that height, while the other delivers almost from the bottom. A stop-cock in this latter is so regulated as to allow the escape of so much liquor as separates by gravitation, while the oil passes away by the other means of exit.—A. R. D.

*Mechanical Apparatus for Charging and Discharging Gas Retorts.* Walker and Bennett, Birmingham. Germ. Pat. 26293, 1883. Dingl. Polyt. Journ., 253 [5], 202.

COAL is fed by means of a feed-screw into the retort B (Figs 1 and 2), which lies in a furnace A. Above the front opening of the retort is a hopper C, which receives the charge of coal, and is closed by a lever and bolt O. At the bottom of it there is the feed-screw R, turning on the shaft F. At the back of the retort there is a pipe L, to conduct the coal-gas to the hydraulic lute. Below L

*Improvements in Methods of Mixing Hydrocarbons with Superheated Steam and Air, and Burning same.* Eng. Pat. 5795. Dec. 18, 1883. R. B. Avery, Washington, U.S.A. This invention received Provisional Protection only.

By this invention the hydrocarbon or oil supply pipe is inserted for a portion of its length in the pipe which supplies the superheated steam, and is fitted with a series of perforated diaphragms of gradually decreasing mesh, so that the oil is atomised, or comminuted, before it enters the steam pipe. Here, in conjunction with the

B Walker und J Bennett's Mechanical Apparatus for charging and discharging Gas Retorts.

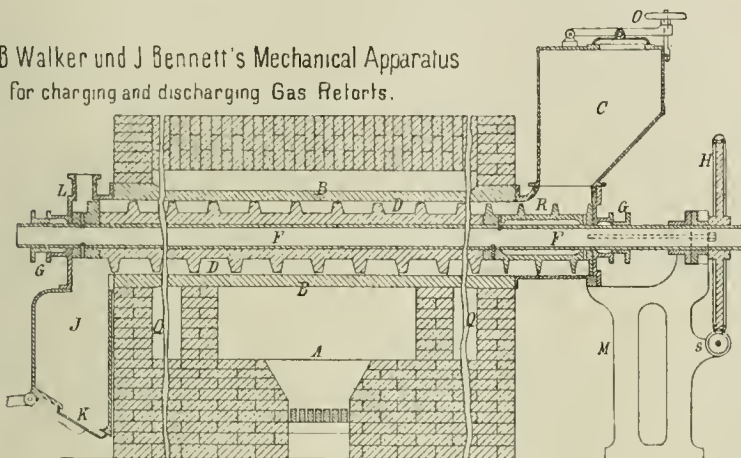


FIG. 1.

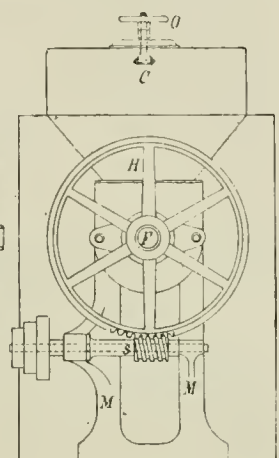


FIG. 2.

is an air-tight box J, to receive the coke. Through the whole length of the retort, and projecting from the stuffing-box G, runs a hollow shaft F, made of cast or wrought iron, carrying within the retort the screw D. The back end of the hollow shaft F is connected with a pipe, which conveys to the furnace the hot air passing through the shaft. The furnace is thus supplied with hot air, and the shaft remains cold.—S. H.

*Gas Producer.* W. Backer, Budweis. Dingl. Polyt. Journ. 253 [5], 203.

THE fireplace G (Figs. 1 and 2) is wider in the upper half than in the lower. By this construction the air cannot travel along the sides. The hot air channel n

steam, it has again to pass through a series of diaphragms similar to the first, so that a thoroughly homogeneous mixture of hydrocarbon vapour and superheated steam is obtained. A short valved branch pipe connects the oil supply pipe with that containing the superheated steam, by which means the oil may both be heated and forced to flow through the first atomising diaphragm. Air may either be forced into combination with the vapour and superheated steam, and so thoroughly mixed by means of the diaphragms, or its supply pipe may be otherwise arranged to secure a perfect mingling of the gas and air at the point of ignition. This mixture is burned in a furnace regenerator of open brickwork, or of some other form suitable for promoting the destructive decomposition of the fuel, and when the brickwork is once heated the current of fuel is for a moment cut off and the flame extinguished. A prompt

W. Backer's Gasproducer.



FIG. 1.

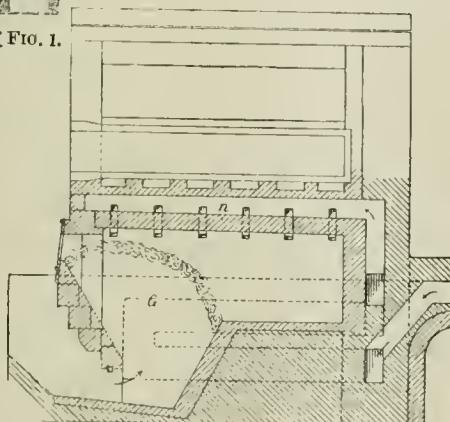
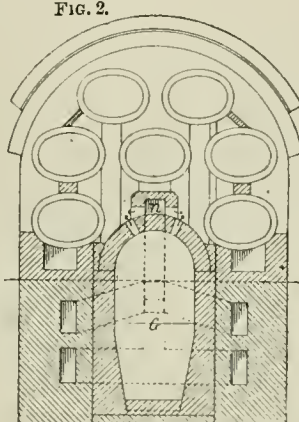


FIG. 2.



rests directly on the vertex of the arch, and here are also the openings for the gas, so that an intimate mixture of air and gas must take place.—S. H.

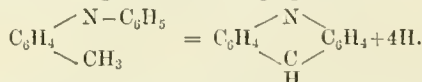
opening of the stopcocks now produces flameless combustion and a heat sufficient to melt iron in a few minutes.—A. R. D.



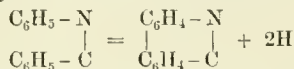
### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Pyrogenous Formation of Acridine.* C. Graebe. Ber. 17, 1370.

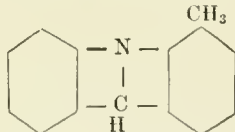
THE synthesis of acridine from diphenylamine agrees more closely with the assumption that its composition is analogous to anthracene rather than phenanthrene, whilst the formation of quinoline, as shown by Ladenburg, is explained more readily when acridine (like phenanthrene) is regarded as a diphenyl derivative. It therefore appeared of interest to the author to see whether it was possible to produce acridine from tolylphenylamine or benzylidene-aniline, analogous to the formation of anthracene from orthobenzyltoluene, or of phenanthrene from stilbene. If acridine is related to anthracene, its formation from orthotolylaniline is possible according to the following equation:—



If, on the contrary, it be regarded as analogous to phenanthrene, benzylidene-aniline must be its source; thus:



The author has passed both aniline derivatives through a tube heated to dull redness, with the result that orthotolylaniline yielded large quantities of acridine, whilst benzylidene-aniline did not produce acridine, but a high boiling base in addition to aniline. In consequence of the small quantity of base yielded its properties could not be studied more closely. This experiment is a further proof of the correctness of Riidel's formula for acridine. From the preceding synthesis and the formation of quinolinecarboxylic acid by the oxidation of acridine it is inferred that acridine is an ortho-derivative. The action of the ditolylamines is similar to that of the tolylphenylamines; thus orthoditolylaniline yields, besides acridine, a methylated acridine of the following constitution:—



It resembles acridine in properties, but has a lower melting point. Paraditolylaniline, however, does not yield a derivative of acridine.—D. B.

*The Purification of Naphthalene by Sublimation.* F. Hirsch. Chem. Zeit. 1884, 839.

EXTENDED experience has decided in favour of a method of distillation for the purification of naphthalene, but since for such process steam is a necessity the author proposes the following plan for obtaining by sublimation a purified naphthalene from the crystalline deposits of heavy coal-tar oil. The sublimation chamber is preferably an isolated structure of masonry, the walls being 30cm. thick, 5m. long, 3m. wide and 3m. high, with a light double roof covered with roofing felt on the exterior and having within a plastered ceiling. At one end of the building is a door, which serves for the withdrawal of the sublimed substance; this door rests in a groove in the frame and during operations is luted in with clay. At the other end, without the main wall, there is a cast iron pan extending across the whole width of the building, set in brick work above a fire the gases of which are led through flues around the pan and thence proceed to the chimney, which rises about 1m. above the roof. Two covers, one of which is dome-shaped, are suspended a short distance above the pan, and act somewhat in the manner of dephlegmators by condensing and returning to the pan any heavy oils which may have volatilised.

An opening in the end wall of the chamber permits of the entry into the latter of the naphthalene vapour, and a wooden lid lined with sheet iron, hinged on the main wall just above the opening, and reaching to the outer edge of the pan, affords a means of filling or emptying the pan. In the roof there is an iron air-pipe projecting 10cm. beyond the ceiling in the interior, and 50cm. above the roof outside. A pan suspended just below this serves to collect any condensed water. The following method of procedure, is recommended. This crude naphthalene which has crystallised out from the creosote oils is allowed to drain for two or three days, whereby it becomes sufficiently dry for a first sublimation. The pan is filled with this crude substance, from 3 to 4 per cent. of slaked lime are added, and after luting down the doors of the chamber, heat is applied, vigorous at first but afterwards somewhat moderated. Samples are taken from the pan at intervals, and when it is observed by the non-crystallisation of a sample on cooling, that the naphthalene has been thoroughly expelled the pan is emptied and charged with a fresh quantity of material. After the sublimation of two or three charges, the chamber is emptied out, and the once sublimed naphthalene is melted in an open cast iron boiler provided with an exit pipe, well mixed first with 20 per cent. of caustic lye of 180° B., and then, after removal of the former, with 6 per cent. of English sulphuric acid of 66° B., with which it is kept in contact for from 15 to 30 minutes. The whole is allowed to stand at rest for about an hour, the acid is then drawn off and the naphthalene freed from acid by washing with water. The washed naphthalene is subjected to sublimation until 80 per cent. has been collected; in this manner there is obtained a white product well adapted for hygienic purposes.—W. D. B.

*Improvements in the Manufacture of Ammonia and Hydrocarbon Gases.* Eng. Pat. 5361. Nov. 13, 1883. W. R. Lake, London. A communication from Thos. Burke Fogarty, Brooklyn, New York.

ORDINARY producer gas, freed from moisture and raised to a high state of incandescence, is led downwards through a tower in which a shower of intimately mixed and finely comminuted carbon and alkali is constantly falling. At the bottom of the tower is a pit containing water to catch the falling material, and provided with an opening for the escape of the gas. A great deal of the nitrogen of the producer gas is taken up by the carbon and alkali to form alkaline cyanides, which are met in their downward course by a steam jet introduced into the lower part of the tower. The effect of this is to decompose the cyanogen compounds with the formation of ammonia, which passes on with the other gas through the aperture at the bottom of the tower. The stream of gas is then led to suitable scrubbers for the recovery of the ammonia, and is freed from aqueous vapour, carbonic acid and carbonic oxide by any method as far as possible. It is next conducted into retorts where ordinary bituminous coal mixed with alkali is undergoing distillation, and is caused to thoroughly permeate the whole mass in the retorts. Here a further combination of nitrogen with carbon and alkali takes place with a further formation of cyanogen compounds. Some of these are decomposed by the watery vapour in the retorts, but the bulk remains in the coal which is left behind in the retorts when the distillation is finished. Another important part is here played by the producer gas. It serves as a diluent or absorbent of the rich hydrocarbon vapours which in the early stages of the distillation of coal are given off in such large quantity that a high percentage of them is condensed into tar for want of a sufficiency of non-illuminant gases, such as hydrogen and light carburetted hydrogen, to dissolve them or retain them in suspension. Atmospheric air or any other nitrogenous gas may be substituted for producer gas in the retort treatment, but producer gas is the most suitable on account of the hydrogen it contains. After leaving the retorts the gaseous products are scrubbed for ammonia in the usual manner. When the distillation is over the coke is brought, with as little exposure to the

air as possible, into a closed vessel, where a stream of water is allowed to play upon it from the top. The steam thus generated decomposes the cyanides with evolution of ammonia, which latter passes upward through the now cool layers of coke above, and, with the other gases generated simultaneously, passes through an opening provided for the purpose. These gases are scrubbed for ammonia, and then, after purification, go to swell the volume of the illuminating gas already obtained from the retorts.—A. R. D.

*Improvements in the Method of Obtaining Volatile Hydrocarbons from Coal and Shale Gas.* Eng. Pat. 5505. Nov. 24, 1883. Edwin Drew, London. (Void by reason of the Patentee having neglected to file a specification in pursuance of the conditions of the Letters Patent).

THIS invention is an improvement on other methods of washing shale or coal gas for the extraction of volatile hydrocarbons. The gas is caused to bubble through or otherwise pass in close contact with heavy oils as in other methods, but the dissimilarity of treatment consists in the gas being in a greatly compressed state during this operation. To remove the heat generated by the compression, it is well to make the passage from the compressing engine to the washers as long as possible. If it is desired to cool the gas below the temperature of the air, this may be done by allowing it to expand before reaching the oil apparatus, as it will still retain sufficient elasticity to cause it to bubble through the column of heavy oil.—A. R. D.

*Improvements in the Manufacture of a Substitute for Bisulphide of Carbon.* Eng. Pat. 5525. Nov. 26, 1883. P. G. W. Typke, W. R. King, and T. T. P. B. Warren, London.

THIS invention relates to the production from petroleum of a substitute for bisulphide of carbon, which can be used for extracting oils and anthracene, for dissolving gums, resins, and analogous substances, for waterproofing and for vulcanising indiarubber. To the distillate from petroleum passing over between 38° and 100° C. is added, with constant agitation, from 2 to 3 per cent. by volume of oil of vitriol. After subsidence the clear liquor is decanted into a still with the addition of from 1 to 2 per cent. by weight of lime or other dehydrating medium, or carbonates, oxides, or metals capable of decomposing the sulpho-oils that may have been generated by the presence of the sulphuric acid. Sometimes the liquid is subjected to repeated treatment with oil of vitriol until the acid ceases to be coloured. The distillation is effected without the injection of steam or water into the still, and the receivers are changed as soon as the distillate reaches a specific gravity of 0.68 or 0.69.—A. R. D.

*Improvements in the Distillation of Coal, Shale, and other Carbonaceous Substances, etc.* Eng. Pat. 5724. Dec. 13, 1883. Peter Conper, Edinburgh, and Martin Rae, of Middleton Hall, Linlithgow.

BELOW each of a bench of retorts is placed a solid fire-brick chamber of such capacity as to hold several charges of the retort above. The distillation of the shale in the retort is preferably carried on at low redness, and effected by external heat through flues surrounding the retort. When the shale has remained here sufficiently long for the more ordinary volatile products to be removed, a portion of the contents of the lower chamber is removed by a door provided for the purpose, to make room for the charge in the retort, which is now allowed to drop below. This underchamber is provided with steam jets in its lower portion so arranged as to draw in air, which promotes the combustion of the coke here. This exposes the charge in the upper portion of this chamber to a temperature slightly higher than that to which it was exposed in the retort. Thus any volatile hydrocarbons which have not been removed by the process in the retort

are here distilled as condensable products. As the charge, now exhausted of volatile hydrocarbons, finds its way downward in the chamber, the carbonaceous matter is burned by the air and steam drawn in as mentioned above, whereby heat is generated for the distillation going on in the upper portion of the chamber, and the nitrogen remaining in the coal is volatilised as ammonia.—A. R. D.

#### IV.—COLOURING MATTERS AND DYES.

*Preparation of Red-Violet Colouring Matters.* Dingl. Polyt. Journ. 252, 343.

(a.) THE Verein Chemischer Fabriken, Mannheim, give the following particulars of the preparation of new colouring matters recently patented by them (Ger. Pat. 26012, 1883):—When  $\alpha$ -naphthol is added to sulphuric acid, maintained at a low temperature, there is obtained, in addition to Schäffer's acid, a new isomeric  $\alpha$ -naphthol sulphonic acid. From this acid, as also from Piria's naphthionic acid, a variety of ponceau colouring matters may be obtained, the corresponding derivatives of Schäffer's acid being orange-red and brown. These colouring matters result from combination with the following diazo-compounds: With diazo-xylene, a ponceau; with diazo-ethylxylene, a blue ponceau; with diazo-azobenzene and its sulphonated derivative and with  $\beta$ -diazo-naphthalene, ponceaux of still bluer shades; with  $\alpha$ -diazo-naphthalene, a deep cherry-red; with diazo-diphenyl (obtained from benzidine), a violet. These colouring matters are all difficultly soluble and extremely fast.

(b.) By the action of carbonyl chloride ( $\text{COCl}_2$ ), upon dimethyl and diethyl aniline, colouring matters are obtained as secondary products, the main reaction consisting in the formation of chloranhydrides and ketone bases. If, on the other hand, a condensing or dehydrating agent, such as aluminium chloride, be also present, the reaction is confined in the main to the production of those colouring matters which belong to the triphenylmethane series. In the case of the dimethyl and diethyl anilines, the colouring matters obtained by this reaction are violets. The process has been patented by the Badische Anilin und Sodafabrik, and they claim its extension to a wide range of the tertiary monamines. (Ger. Pat. 26016, 1883).—C. F. C.

*Auramine, a New Yellow Colouring Matter.* Dingl. Polyt. Journ. 253, 86, 1884.

THIS colouring matter, introduced by the Badische Anilin und Sodafabrik on the one hand, and by Bindschedler and Busch upon the other, has been received with considerable favour, and is interesting in that it is the first artificial yellow dyestuff which can be fixed upon vegetable fibres in the same manner as the basic aniline colours—by the aid of tannic acid. According to C. Köchlin, a steam colour is prepared by mixing auramine with its own weight of tartaric acid and six times its weight of tannin. After printing with this mixture upon a vegetable fabric which has not been prepared with tin, and steaming, the fixation of the colour may be completed by the usual treatment with tartar. The tint varies from golden to sulphur or citron-yellow, according to the quantity of dyestuff employed. Auramine yellow resists the action of soap and light, but is somewhat readily altered by chlorine; it dyes wool of a very pure tint, and on account of its property of being fixed by tannin may be advantageously employed in conjunction with other tannin colours to obtain, for instance, a malachite green with yellowish tone. The mordanting of cotton fabrics with oil appears, so far as auramine is concerned, harmful rather than otherwise. This new yellow will probably, to a considerable extent, take the place of Persian berries: not on account of superior fastness or depth of tone—for in respect of fastness the latter yield a tint comparable with the alizarin colours—but rather on account of its



price. The first step to replace Persian berries by a less expensive artificial yellow was taken by A. Poirrier when he introduced his *jaune solide*, an azo-colouring matter fixed by the aid of acetate of chromium. As this, on steaming, behaved with the basic aniline colouring matters like the dye from Persian berries, it was found valuable in the production of mixed tints. By itself, however, *jaune solide* gives an orange-yellow tint, which resists the action of soap and light very well, but lacks that purity of tint which is characteristic of auramine. A third new yellow colouring matter, only applicable, however, for silk and wool, is flavaniline, introduced by Meister, Lucius and Brünig. It yields tints equal for purity and fastness in every respect with auramine; according to Köchlin, it is best fixed, in printing, by its own weight of tartaric acid and acetate of magnesia. (See also *Journ. Soc. Chem. Ind. Abstr.*, p. 318, 1882).—W. D. B.

#### On Canarin. *Dingl. Polyt. Journ.* 253, 130.

MESSRS. DURAND and HUGENIN offer by the name of canarin a yellow dyestuff, which is obtained by the action of Cl or Br on sulphocyanic acid. Prochoroff and O. Miller publish the following method for preparing it:—Mix 370cc.  $\text{SO}_4\text{H}_2$ , 660cc. HCl and 1350cc.  $\text{H}_2\text{O}$  and add gradually an intimate mixture of 1kg. CNSK and 500gr.  $\text{KClO}_3$ , taking care that the temperature does not rise above  $60^\circ\text{C}$ . Or dissolve 1kg. CNSK in 1ltr.  $\text{H}_2\text{O}$ , add 20cc. HCl or HBr, and then very slowly 1kg. Br; cool all the while. Some gas is given off and an orange precipitate is deposited, whose weight is about 40 per cent. of the CNSK employed. Washed with water, it is crude canarin. By dissolving it in KOH and adding alcohol, a potash compound is obtained, which, on decomposing with HCl, gives the pure canarin. Dried at  $100^\circ\text{C}$ . it forms a reddish-brown, strongly glittering powder, insoluble in  $\text{H}_2\text{O}$ , alcohol and ether, and soluble in  $\text{SO}_4\text{H}_2$  and KOH. H. Schmid believes it to be persulphocyanogen,  $\text{C}_3\text{N}_3\text{S}_3\text{H}$ . O. Miller, however, contends that canarin dissolves in  $\text{SO}_4\text{H}_2$ , giving off  $\text{SO}_2$ ; whereas  $\text{C}_3\text{N}_3\text{S}_3\text{H}$  does not reduce  $\text{SO}_4\text{H}_2$ . To employ the canarin of the market for dyeing, dissolve 1 part canarin in 1 part KOH and 20 parts  $\text{H}_2\text{O}$  at a slight heat, add 7 to 10 per cent. soap to the brown solution and allow to cool. Water containing CaO or MgO must be avoided, as canarin precipitates these bases. NaOH cannot be taken in lieu of KOH, the sodium compound being insoluble in the cold. Too long heating with KOH decomposes the dyestuff. 80ltr.  $\text{H}_2\text{O}$  are then mixed with 60ltr. canarin solution, and the goods drawn through the cold solution, pressed, wound up on a roller and allowed to remain for some hours in the wound up state. They are then washed and soaped. H. Koechlin has improved Prochoroff and Miller's process for dissolving. He boils 100gr. canarin and 100gr. borax with 1ltr.  $\text{H}_2\text{O}$ . The dyeing is afterwards performed similar to alizarin dyeing by gradually raising the temperature. On adding a thickening to the canarin solution in borax a pure yellow can be printed. Cloth dyed with canarin resists light and soap extremely well. Its low price and the readiness with which it can be employed give it a fair chance of success. Its property of acting as a mordant for basic aniline dyes is also important, and Koechlin, by dyeing methylene-blue, malachite-green, and Paris violet on canarin, obtained colours which resist warm soap solution.—S. H.

*Diresoreinol and Diresoreinphthalcin*, by R. Benedikt and P. Julius. *Monatsh. f. Chem.* 177, 1884.

THE products of the fusion of resoreinol with sodium hydrate have been shown by Barth and Schreder (*Berl. Ber.* 12, 503) to be phloroglucol (60-70%) and diresoreinol (1-5%). This is confirmed by the authors' experiments. When, however, the fusion is carried out with large quantities of the reagents, as it has been by Merck, of Darmstadt, the proportion of the diresoreinol is largely increased. The authors have extracted in this case from

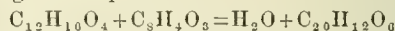
residues, left after extracting the phloroglucol with ether, from 25-30% of diresoreinol. It is best isolated by crystallisation from water. It melts without decomposition at  $310^\circ$ .

*Acetyl diresoreinol*.— $\text{C}_{12}\text{H}_8(\text{O}.\text{C}_2\text{H}_5)_4$  is prepared by boiling the anhydrous diresoreinol with acetic anhydride and sodium acetate. It crystallises from alcohol in shining prisms (m.p.  $152^\circ$ ).

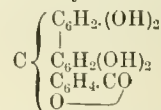
*Hexanitrodiresoreinol*.— $\text{C}_{12}(\text{NO}_2)_6(\text{OH})_4$  is the product of the action of fuming nitric acid upon the phenol. It is characterised by its solubility in water. It is decomposed at  $230^\circ$  without previously melting.

*Decabromodiresoreinol*.— $\text{C}_{12}\text{Br}_6(\text{OBr})_4$  was obtained by dissolving the phenol in aqueous potash, and adding a solution of bromine in concentrated hydrochloric acid. This compound gives off bromine at the ordinary temperature; between  $170^\circ$  and  $185^\circ$  it loses approximately  $2\text{Br}_3$ , and is converted into an insoluble product, which latter, on treatment with  $\text{SO}_2$ , yields hexabromodiresoreinol.

*Diresoreinphthalein*.—The condensation of these diphenols with phthalic anhydride throws light upon the constitution of the phthaleins. The authors find that in the case of diresoreinol this reaction takes place according to the equation—



the soluble phthalein which results having therefore the constitution represented by the formula:



By the reaction, on the other hand, of the diphenol with phthalic anhydride in the proportion of two mols. to one mol. of the latter, an insoluble phthalein,  $\text{C}_{32}\text{H}_{20}\text{O}_8$ , is produced. It separates from its solution in alcohol as a heavy crystalline powder. This body dissolves in a concentrated solution of potassium hydrate, with development of a blue colour which disappears on dilution, becoming violet on boiling, and again disappearing on cooling. The soluble phthalein,  $\text{C}_{20}\text{H}_{12}\text{O}_6$ , had been already obtained by Link (*Berl. Ber.* 13, 1654). The authors prepared the substances according to his method, and obtained a body identical in properties. The bromine derivative of this phthalein approximated in composition to  $\text{C}_{26}\text{H}_8\text{Br}_4\text{O}_6$ , which confirms the constitutional formula assigned by the authors to the phthalein.—C. F. C.

*Origin and Preparation of Annatto*. P. Vieth. *Chemisches Centralblatt* 15, 411.

THIS colouring matter, largely used for colouring butter and cheese, occurs in the annatto tree (*Bixa Orellana*). The fruit capsules of this tree contain a large number of seeds embedded in pappy reddish coloured fruit-flesh, similar to that surrounding the seeds of the ripe love-apple. Annattoin is obtained by macerating the fruit-flesh or colouring matter in cold water. It is largely employed now-a-days for dyeing and printing woollen and cotton goods, the colour being fast and fine. Basket or paste annatto is prepared in the tropics by placing the seeds in large vessels of water and converting the whole by prolonged heating into a thick pap. By this process the brilliancy of the colour is greatly impaired, so that basket annatto cannot with advantage be used for dyeing fabrics, also cheese coloured with it gradually fades. The colouring matter is used by the natives of Brazil to flavour their food.—F. L. T.

#### V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Behaviour of Coloured Fabrics to Water and Air*. By S. Bounhoff. *Chemisches Centralblatt* 15, 460.

EXTENDED experiments lead to the conclusion that the colour of the goods has no influence either on the amount or rate of absorption of the hygroscopic water. The degree of evaporation of the capillary water is the same

in coloured and non-coloured goods, but the evaporation does not proceed so regularly in the case of the non-coloured as with the coloured goods. The colour is of considerable influence on the permeability of the goods to air.—F. L. T.

*The Manufacture of Textile or other Fabrics or Materials coated or covered with Xylonite, Pyroxyline or similar substances, which invention comprises various articles made with the said materials, and apparatus for use in such manufacture.* Eng. Pat. No. 3554, Nov. 27, 1883. Henry Harris Lake. A communication from Jarvis Bonesteel Edison.

The Xylonite or similar substance is made into a very thin sheet by cutting, as in the process of making veneer. The thin sheet and the material to be coated are then simultaneously passed through a pair of pressure rollers, one surface of the sheet having been treated with a solvent immediately before contact. In this way waterproof materials can be obtained applicable to many purposes. By introducing a polished plate, say of glass, between the rollers, and with its surface in contact with the thin sheet of xylonite, a very bright and highly polished surface is produced on the latter. Devices can be readily produced on the material by suitably engraving the glass plate. A very highly polished imitation patent leather can be obtained by coating sheets of leather with xylonite. The process can be applied to a great number of useful purposes in the arts. Descriptive drawings accompany the specification.—E. J. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

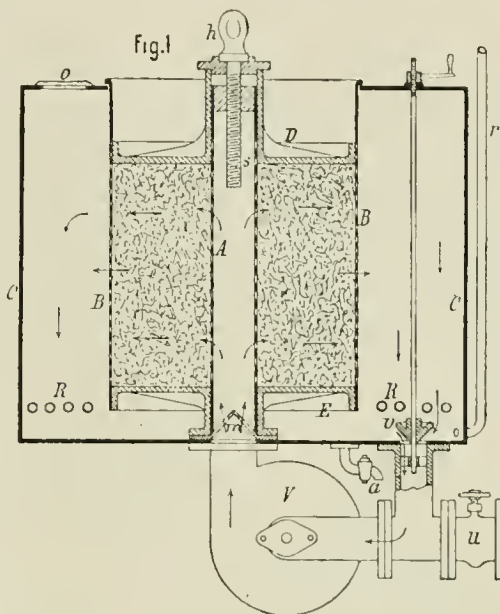
*Abridged process for Turkey-red Dyeing and Alizarin Printing.* A. Müller. Dingl. Polyt. Journ. 252, 219.

It is known that a number of the volatile organic ammonia bases produce precipitates with solutions of alumina and tin salts, which dissolve in an excess of the precipitant (e.g. mono- di- and tri-ethylamine, the butyl- and amyl-amines and some di- and tri-amines). The author utilised this property experimentally, in order to combine the mordanting of the fabric with oil and alumina in one operation, whereby the chalk bath, necessary at other times, is omitted, the fabric being ready to receive the alizarin after it has been dried and the base removed by volatilisation. A clear saturated solution of aluminium hydroxide in 10 p.c. ethylamine was treated with about 15 p.c. Turkey-red oil, also neutralised with ethylamine. The fabric, after being boiled and dried, was treated with the clear mordant and dried at the ordinary temperature. It was then dyed with alizarin in the usual manner and soaped, the result being the development of a bright red colour. The author thinks that if it were possible to prepare ammonias of the above character cheaply on a large scale the success of this method would be established, especially if by some suitable arrangement the volatile bases employed could be recovered.—D. B.

*Improvements in Dyeing, Bleaching, etc.* Dingl. Polyt. Journ. 253 [3] 126.

CERRUTTI-SELLA's process of exposing fibres and cloth for dyeing, bleaching, washing, etc., to the action of a circulating stream of liquor, has lately again been brought forward in the shape of a machine constructed by O. Obermaier (Germ. Pat. 23117, 1882). It consists of a drum B (Fig. 1) with perforated sides, in the centre of which is a similar but much narrower drum A provided with larger holes. The material for treatment is put in the space between the drums, the lid D screwed down firmly, and the whole placed into a cylindrical vessel C in such a manner that the bottom opening of A rests on the delivery pipe M of a pump V. The cylinder C is also connected with the pump V by a valve *v* which can be opened and shut at pleasure. To commence the operation the necessary liquor is forced through

*u* into the drum A; it then penetrates the stuff, passes through the holes of B and runs into C. After some time, when the liquor in C has attained a certain height, *u* is shut off and *v* opened. The liquor now moves in a



cycle. To bring about its uniform distribution care is taken to press down the material to be treated as firmly as possible. The holes of the two drums are of a different size, which prevents the formation of channels for

Osw Fischer. (2a3)

Fig 2

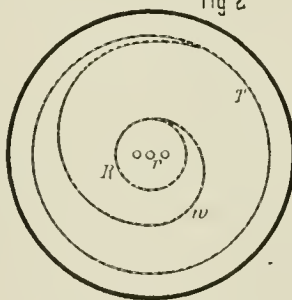
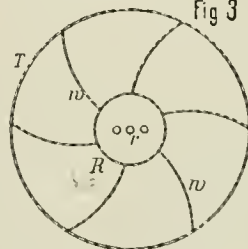


Fig 3



the passage of the liquor. This apparatus can serve for washing, scouring, dyeing, bleaching, mordanting, souring, oiling, &c., of loose fibres, cloth, furs, felts, ribbons, and so forth. During succeeding operations as (e.g. for wool) during washing, carbonising, dyeing, drying, &c., the stuff remains in the pressed state in the same place, and only the drum as a whole is shifted by means of a



crane into different vessels C. The principal cause of felting is thus removed. For drying, the pump is replaced by a fan blowing in warm air. The apparatus may also be arranged in such a manner that the liquor is forced from C into B and sucked from here into A, passing through the pump and returning to C. Or better still, the whole may be made to turn round *m* like a centrifugal machine. The centrifugal force causes the liquor to penetrate the material, pass to C, whence it is pumped again into A and re-enters the cycle. In connection with this patent O. w. Fischer (Germ. Pat. 22674, 1882) has devised a contrivance to prevent the too quick passage of the liquor through the material. As Figs. 2 and 3 show, the space between the drums is partially taken up by perforated spiral plates or bent perforated plates, dividing, as it were, the whole space in several distinct compartments. The liquor enters through the pipe *r* and by having to make a longer way it is compelled to remain in contact with the material for some time. To bring about uniform dyeing all over a piece of cloth, the Société anonyme des teintures et apprêts de Tarare (Austr. Pat., 1883), makes dye liquor to run along a slanting board *F* (Fig. 4) from a trough C, which is kept

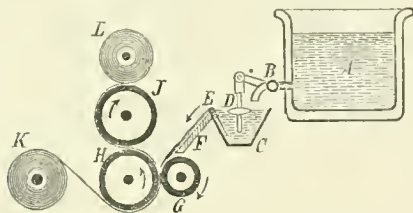


FIG. 1.

at the same level by a self-regulating tap B. The cloth is unwound at K, goes over the roller H, receives here at G the liquor, then proceeds over J and is wound up at L. The rollers H and J are hollow, and can be heated by steam. By altering the speed of the cloth and the flow of the liquor every shade of colour can be attained.—S. II.

*On the application of Chlorine-gas as a discharge in Calico-Printing.* Alb. Scheurer. Dingl. Polyt. Journ., 253 [5] 208, and Bull. Soc. Ind. Mulhouse, 1884, 364.

PERSOZ' suggestion to discharge dyed indigo-blue by means of Cl gas has not been adopted by the trade owing to the time the Cl requires to destroy the colour, even in the presence of  $H_2O$ . To produce white on a blue ground, the Cl must act on the damp spots for at least two minutes: however, this time is also sufficient to give the blue a greenish tint. The author has found that a cloth dyed indigo-blue and steeped in a weak NaOH solution is completely decolorized by Cl gas in 10 seconds. Caustic soda lye of 16° to 17° B. acts best. In this manner the most powerful oxidation is brought about, which no dye-stuff can resist. Turkey-red turns yellow in 20 seconds, and gives a pure white in 50 seconds. Aniline-black is destroyed almost instantaneously. Cloth steeped in a mixture of aniline-oil and NaOH, and exposed to the action of Cl gas, turns black at once. A solution of an aniline-salt produces under the same circumstances a bright-yellow which resists well even the action of Cl. If a drop of NaOH is brought on a spot of such cloth and Cl allowed to act again, the colour is at once destroyed in that particular spot. The hypochlorites, even in concentrated solution, do not decolorize indigo-blue at once, whereas the free acid acts like Cl. But it cannot be free HClO which plays the important part in this process, as there is always present alkali in excess to saturate the free acid, and the stuff, indeed, has an

alkaline reaction after the destruction of the colouring matter. The agent is probably O *in statu nascendi*, thus:  $2NaOH + 2Cl = 2NaCl + H_2O + O$ . By this process the discharge of indigo-blue and mordanting with  $Al_2O_3$  for alizarin-dyeing can be effected at the same time by using a mixture of NaOH and  $Al_2O_3Na_2$ . To produce chrome yellow in discharge-style a mixture of PbO and  $Cr_2O_3$  in NaOH is exposed to Cl gas. White is produced by simply printing on NaOH and a thickening and exposing to Cl gas.—S. II.

*Bleaching and Scouring of Fibres.* H. Köchlin, Lörrach. Germ. Pat. 27745, 1883.

THE inventor bleaches fibres by treating them with alkalis or hydrates of alkaline earths, and heating them afterwards by steam or hot air. But as the steam is never free from air, the fibre comes in contact with O, whereby it is oxidised. To prevent this oxidation, which would injure the strength of the fibre, a reducing agent, viz.,  $Na_2SO_3$  or  $NaHSO_3$ , is added to the caustic solution. Cotton, &c., is therefore first steeped in boiling water acidified with  $SO_2H_2$  to remove starch-like matters, then plunged into a mixture of NaOH and  $NaHSO_3$  and pressed and heated for an hour. The  $NaHSO_3$  to be added is in proportion to the air contained in the steam.—S. II.

## VII.—ACIDS, ALKALIS, AND SALTS.

*The Non-existence of Ammonium Hydrate.* By D. Tommasi. Chemisches Centralblatt, 15, 418.

J. THOMSEN, from physical and thermochemical considerations, concludes that this hydrate does not exist. The author has come to the same conclusion in a different way, for on calculating all the heats of formation of the soluble hydrates and comparing them with the observed, a close agreement is found except in the case of ammonium:—

	Heat of combination.	
	Calculated.	Observed.
Sodium hydrate .....	77.7	77.6
Lithium ..	83.4	83.3
Thallium ..	20.4	20.0
Calcium ..	150.6	150.1
Barium ..	28.1 + x	28.0 + x
Strontium ..	158.6	158.2
Ammonium,, ..	54.2	21

It follows that the constitution of ammonia solution must be different from that of the alkaline hydrates, and accordingly it is wrongly compared with potassium and sodium hydrates.—F. L. T.

*Occurrence of Valerolactone in Wood Vinegar.* M. Grodski. Ber. 17, 1369.

SOME time ago the author, in conjunction with Krämer, gave a description of the acids which occur in the high boiling fractions of wood vinegar, as well as in the uncrystallisable liquors obtained as by-product in the preparation of sodium acetate from wood vinegar, and crude acetic acid from calcium pyrolignite. Starting from the same source, the author has recently isolated

a body which has hitherto not been known to exist in wood vinegar. From its properties and composition it was recognised as the lactone of the normal valeric acid. It represents a colourless liquid, which solidifies at  $-18^{\circ}$ , boils at  $206^{\circ}$ , and dissolves in water forming a neutral solution, from which potassium carbonate separates the lactone in an unaltered condition. On oxidation with chromic acid succinic and acetic acids are obtained. The sp. gr. of the new substance is 1.0831 at  $20^{\circ}$ . On analysis numbers corresponding with the formula  $C_5H_8O_2$  were obtained.—D.B.

*The Sulphuric Acid and Alkali Trade of England.\**  
Dr. G. Lunge, Chem. Industrie, 1884, 213.

THE author gives some statistical information on the alkali trade which is of considerable interest. The figures were chiefly indirectly supplied to him by the Hon. Secretary of the Alkali Makers' Association. Recourse was also had as far as possible to blue-books.

*The total import of Pyrites and Brimstone was as follows:*  
(Official Publications of the Board of Trade.)

TABLE I.

Year.	a Import of Pyrites.	b Burnt Ore worked up for Copper.	c Cupreous Pyrites burnt, calculated from b.	d Import of Brimstone.
	Tons.	Tons.	Tons.	Tons.
1872	516,299	253,529	362,184	—
1873	520,939	323,910	462,729	—
1874	500,831	329,004	470,006	51,821
1875	539,256	365,368	521,954	55,876
1876	505,301	379,269	511,813	13,735
1877	680,033	427,954	611,363	54,167
1878	577,719	402,716	575,308	43,604
1879	481,622	385,874	551,250	41,012
1880	657,867	415,367	593,670	46,896
1881	542,046	396,737	566,767	10,561
1882	626,902	431,427	620,610	47,278
1883	600,673	439,156	627,365	43,882

Column *a* shows the total import of pyrites, but it would be wrong to calculate from these figures the amount of sulphuric acid manufactured, as it is well known that the stock of pyrites in hand, of the companies, is subject to considerable fluctuations. However, column *b* supplies the want for all practical purposes, and if it be granted that 70 parts of burnt ore correspond to 100 parts of pyrites, column *c* is obtained on which to base further calculation. This column also shows, that for the last 10 years nearly all the pyrites burnt consisted of the so-called "copper-pyrites."

Another table taken from the "Mineral Statistics of Great Britain and Ireland" for 1882, p. 84, shows the origin of the pyrites, thus:—

\* For brevity' sake the expression "England" always implies Great Britain and Ireland.

TABLE II.

Pyrites imported from

Year.	a Norway.	b Portu'l.	c Spain.	d Germ'y.	e Other Countries	f Total.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
1873	67,462	119,559	246,692	—	6,634	520,347
1874	41,044	162,569	291,117	—	907	498,637
1875	21,820	165,433	311,019	—	6,283	537,555
1876	7,688	56,579	419,068	—	21,417	504,752
1877	8,564	119,562	498,977	—	22,209	679,312
1878	5,773	136,705	419,561	—	12,318	579,261
1879	8,485	82,529	374,505	—	15,837	481,392
1880	10,952	166,519	463,199	8,695	8,684	658,017
1881	6,009	110,079	379,216	8,412	8,662	542,378
1882	—	111,132	497,807	—	15,761	627,700

The import of 1882 was distributed to the principal ports of this country as follows:—

To Liverpool.....261,085 tons.  
To London.....49,912 "  
To the Tyne district.....131,952 "  
To Glasgow.....25,520 "

The production of pyrites mined in England is small (25,400 tons in 1882), and as the ore is very poor in sulphur, it is principally used for coppers and rouge making. Some Irish pyrites are probably employed in Ireland for sulphuric acid manufacture.

Table III. shows the amount of sulphuric acid manufactured, expressed as monohydrate,  $SO_4H_2$ : O.V.

TABLE III.

Year.	a Acid made from Pyrites.	b Pyrites Acid used for Salt-cake manufcture	c Pyrites Acid otherwise employed.	d Acid made from Brimstone.	e Total.
	Tons.	Tons.	Tons.	Tons.	Tons.
1878	747,900	417,406	330,491	124,271	872,171
1879	716,625	445,265	271,360	116,881	833,569
1880	771,771	501,612	270,159	133,653	905,421
1881	736,797	475,724	261,073	115,599	852,396
1882	806,793	472,151	334,642	131,742	911,535
1883	815,574	484,252	331,322	125,064	940,638

Column *a* is calculated from Table I. *c*. The pyrites are supposed to contain 48% S, of which 45% S are burnt in the kilns. 100 parts of pyrites yield 130 parts of  $SO_4H_2$ . Column *b* shows the quantity of pyrites-acid for salt-cake manufacture. The amount of NaCl used for saltcake making being known (Table V.) and suppose 7 NaCl require 4 pyrites, and 100 pyrites yield 130  $SO_4H_2$ , then 100 NaCl require  $7 \cdot 3 SO_4H_2$ . Column *c* gives the amount of oil of vitriol (O.V.) used for other purposes, chiefly for the preparation of artificial manure. Column *d* shows the quantity of O.V. made from brimstone\*  $100S = 285 SO_4H_2$ . The amount appears very large

\* It is supposed that the amount of sulphur made in this country is equal to that used for gunpowder making, bleaching of wool and silk, etc., that is to say, it does not serve for sulphuric acid manufacture.



and can only be accounted for by the fact that sulphate of ammonia makers, bleachers, tar distillers, etc., employ exclusively such acid on account of its being free from Fe and As. It is worth while to point out here that German makers of sulphate of ammonia use pyrites-acid and Professor Lunge terms the English system of exclusively employing brimstone acid for the same purpose, "*prejudicial*."

Table IV. gives the statistics of the alkali industry proper.

worthless by-product no longer. Professor Lunge thinks that it is unimportant whether the ammonia soda maker will ever succeed in making HCl from his  $\text{NH}_4\text{Cl}$  or not, because this operation must certainly be more expensive than the production of HCl in the Leblanc process, which, after all, is unavoidable. The price of the soda of the Leblanc process must conform to the price of that of the ammonia process, which undoubtedly works cheaper; on the other hand, the HCl of the latter process, if it can be recovered at all with advantage, must conform to the

TABLE IV.

Year.	<sup>a</sup> Ammonia Soda 48°	<sup>b</sup> Calcd Leblanc Soda, 48°	<sup>c</sup> Caustic Soda 60°	<sup>d</sup> Soda Crystals 21°	<sup>e</sup> Bicarbonate 38°	<sup>f</sup> Total Leblanc Soda, 48°	<sup>g</sup> Total Soda 48°	<sup>h</sup> Total Soda expressed as 100% $\text{Na}_2\text{CO}_3$ .
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
1878	10,000	197,992	84,612	170,872	11,756	387,808	397,808	326,203
1879	18,000	228,209	86,511	185,319	13,083	427,837	445,837	365,586
1880	26,000	258,893	106,381	192,926	13,539	486,882	512,882	420,563
1881	34,000	225,087	108,310	203,773	12,853	459,856	493,856	404,962
1882	42,000	230,213	116,861	180,846	11,115	466,562	508,562	417,021
1883	52,000	228,034	119,929	188,678	13,609	471,220	523,220	429,040

This table is extremely interesting in as far as it shows that the production of Leblanc-soda of every kind has not diminished, as is often asserted, but is as high as it ever was. It was to be expected that the total consumption of soda would increase with the progress of civilisation. This increase has, indeed, taken place, but whereas the production of Leblanc-soda remained stationary, that of ammonia-soda has increased *fivefold in six years*. The "victorious advance" of the ammonia process was also accompanied by a steady fall of prices, and so it came to pass that altogether 20 alkali works—

price of the HCl of the former, which can certainly be made a great deal cheaper. Mr. Mond's very ingenious process is of very restricted application, and is of no consequence at present. (See Mr. Weldon's address in the July number of this Journal.) Mr. Weldon's beautiful idea of combining Leblanc-soda and coke making in order to recover and make use of the by-products will be of no practical value, as the recovery of these will not cost less, but probably more than they are worth, when the loss of calorific-value is taken into account. (See calculations of Mr. Schenrer-Kestner. *Compt. Rend.*, vol. 97, 179.)

TABLE V.

*Consumption of NaCl and the production of Bleaching Powder.*

YEAR.	<sup>a</sup> NaCl used in the Alkali Industry.	<sup>b</sup> NaCl equal to Ammonia Soda produced.	<sup>c</sup> NaCl used for Salt Cake Manufacture.	<sup>d</sup> NaCl converted into Leblanc Soda.	<sup>e</sup> NaCl used to make S. C. for Sale.	<sup>f</sup> Quantity of Bleach made.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
1878	568,542	12,000	556,542	465,370	91,172	105,044
1879	645,287	21,600	593,687	513,104	80,283	115,290
1880	700,016	31,200	668,816	584,258	84,558	131,606
1881	675,099	40,800	634,299	551,827	82,472	135,826
1882	679,935	50,100	629,535	559,874	69,661	135,170
1883	708,070	62,400	645,670	565,461	80,206	141,868

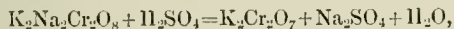
four at Widnes, two at St. Helens, one at Manchester, 12 on the Tyne, etc.—had to discontinue their operations. The table shows at the same time that the deficiency caused by the stoppage of these works must have been made up by the increased output of the remaining works. Quantitatively no decrease in Leblanc soda making has taken place.

Owing to the combination of the bleaching powder manufacturers, the price of bleach has lately risen considerably. The HCl of the salt-cake manufacture is a

Column *a* is not absolutely correct, as the ammonia process requires two parts of NaCl for one part of  $\text{Na}_2\text{CO}_3$  produced. However, for the sake of comparison, and in order to obtain columns *b* and *c*, the author calculates that 120 NaCl yield 100  $\text{Na}_2\text{CO}_3$  of 48 degrees in either process. Column *f* shows the production of bleach, which has not fallen off in spite of the combination among the manufacturers; nay, it is even larger in 1883 than ever before. However, it is very probable that in 1884 we shall see some decrease.—S. H.

*Process for Preparation of Potassium Bichromate*  
P. Römer, *Chemisches Centralblatt*, 15,352.

THE author attacks 100 pts. chrome ore with 150 pts. lime, 40 pts. potash, and 30 pts. soda. The melt is lixiviated and the lye treated with sulphuric or hydrochloric acid, according to the equation—



whereupon the potassium bichromate separates out, the sodium sulphate remaining in solution and being recovered by evaporation.—F. L. T.

*Calcium Hydrosulphides.* By Edward Divers, M.D., and Tetsukichi Shimidzu, M.E., *Chem. Soc. Journal*, July, 1884.

THE authors have succeeded in obtaining for the first time solid calcium hydrosulphide. The preparation is exceedingly tedious, and many precautions are necessary, it being absolutely necessary to exclude air during the formation and crystallisation. Their method was as follows:—A glass tube 20mm. in diameter and 1½ decimetres in length was drawn out at one end to a narrow tube, which was then bent twice at right angles, and connected with a sulphuretted hydrogen apparatus. One part of lime, prepared by igniting precipitated calcium carbonate, was added to four parts of hot water contained in the tube, and sulphuretted hydrogen passed in. The other end of the tube was connected with a wash-bottle, to prevent access of air. Owing to the very great solubility of calcium sulphide it was necessary to make further additions of lime as each lot dissolved. The concentrated solution thus prepared was warmed slightly to redissolve the crystals that had formed, and left to clarify by subsidence. It was then decanted and placed in ice, when it yielded an abundant crop of crystals of calcium hydrosulphide. Calcium hydrosulphide forms colourless prismatic crystals which melt with slight decomposition in their water of crystallisation on a slight rise in temperature. It appears to be soluble in less than one fourth its weight of water. An almost saturated solution, even when warm, loses only a trace of water in a current of dry hydrogen sulphide. The analyses made gave excellent results, considering the difficulty in handling such a substance. It crystallises with 6 mols. of water. The authors have also obtained calcium hydroxyhydrosulphide by the action of calcium hydroxide on calcium hydrosulphide, in the form of colourless four-sided prisms. Calcium monosulphide was prepared as a white amorphous solid by the action of hydrogen sulphide on crystals of calcium hydrosulphide. The latter part of the paper is taken up with an exhaustive consideration of the reactions and decomposition of various sulphur compounds of calcium.—E. J. B.

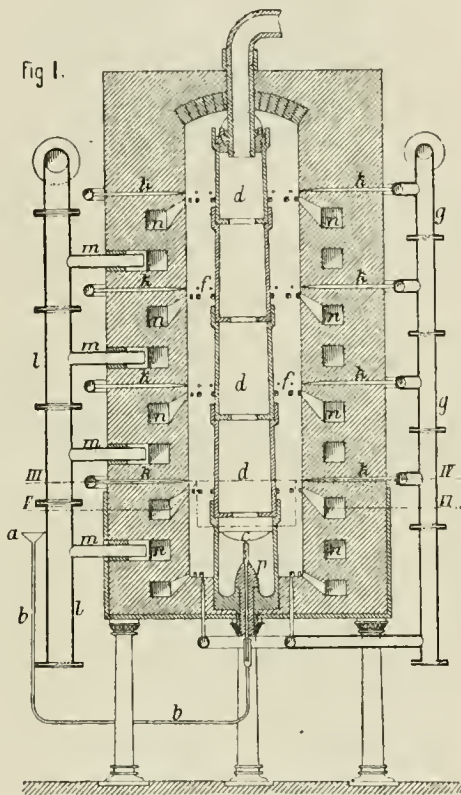
*Improvements in Means and Apparatus for the Treatment of Alkaline Salts at high temperatures, applicable also to other uses.* Eng. Pat. No. 5489, Nov. 22, 1883. Camille Alphonse Faure.

THIS invention is an improvement on that patented by M. Faure, in 1882, No. 6058. Among other purposes for which it is applicable is the preparation of metallic sodium from carbonate of soda. The apparatus consists of a furnace or vessel composed of some refractory material, such as compressed magnesia. Embedded in the bottom of the vessel arc electric conductors made of any suitable material. Suitable openings are made in the vessel for the supply of material, the escape of gas, and the vapour of sodium. A hole is placed in the bottom for the discharge of scoria or metals, such as aluminium. The vessel itself may be made a conductor of electricity. The apparatus is heated by a powerful current of electricity, though for the sake of economy it sometimes receives a preliminary heating by means of waste gases. The inventor has succeeded in making tubes of compressed magnesia, a thing hitherto unknown. The specification is accompanied by drawings.—E. J. B.

*Apparatus for Decomposing  $\text{SO}_4\text{H}_2$  in the Manufacture of  $\text{SO}_3$ .* H. Angerstein, Schalke. Ger. Pat. 26,959, 1883.

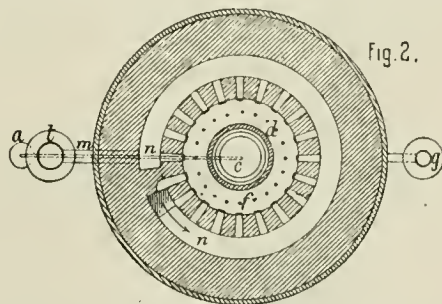
IN Winkler's process the vertical decomposing pipe is rapidly destroyed by  $\text{SO}_4\text{H}_2$  dripping on to it from above. The patentee has devised an apparatus where the  $\text{SO}_4\text{H}_2$  is admitted from below and evaporated in a shallow platinum dish. The decomposing pipe (Figs. 1, 2, 3)

fig 1.



consists of a number of small socket pipes *d*, made of fire-clay, and put one above the other. Each is provided with a perforated bottom on which to pile up lumps of

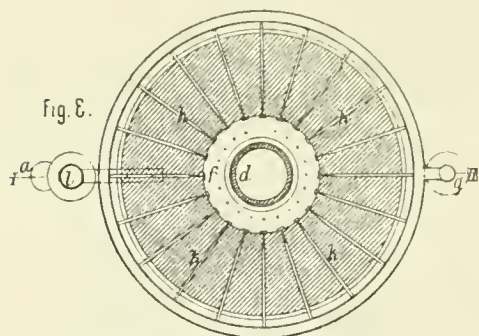
fig. 2.



broken clay. A porcelain nozzle *p* projects into the lowest pipe from below, and a platinum tube *b* passes through it. This tube carries at the top a platinum dish *c*, and to the bottom is screwed a thin platinum tube *b*, twice bent at right angles. The furnace proper is made of fire bricks, and through its sides and bottom pass a number of narrow channels, *k* and *m*, which serve to introduce producer gas and air—both under pressure—from two separate mains. The air is heated by passing through hot flues and a very high temperature is produced. The



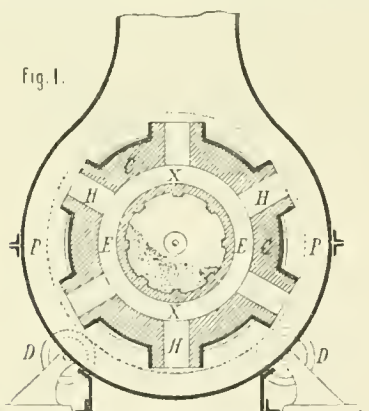
$\text{SO}_2\text{H}_2$  runs continually into the funnel *a*, and thence through *b* into *c*, where it is evaporated. The vapours



are decomposed on their way in  $\text{SO}_2$  and  $\text{O}$ , and then carried to the condenser, and over platinised asbestos for the conversion into  $\text{SO}_3$ .—S. H.

*Manufacture of Chlorine.* W. Weldon, London. Germ. Pat. 27137, 1883. Dingl. Polyt. Jour. 253 [4] 156.

To oxidise a mixture of  $\text{MnCl}_2$  and other substances the author uses a cylindrical retort which is heated from outside, muffle-fashion, and allows of the introduction of the charge automatically at one end, and of its withdrawal

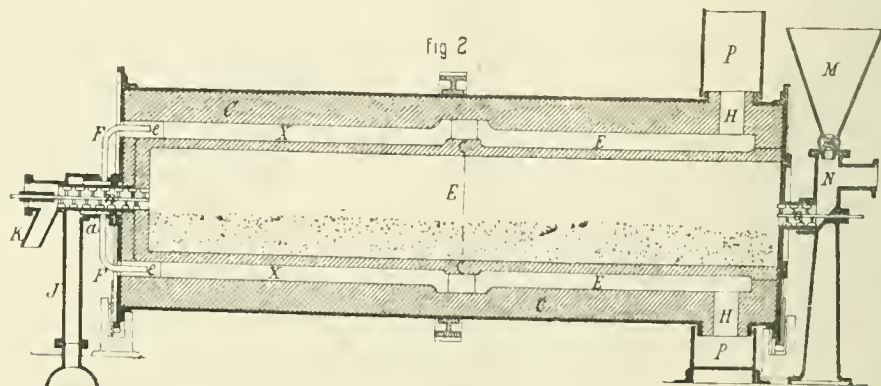


at the other end as a solid mass. The retort *E* (Figs. 1 and 2) is made of fire-clay or cast-iron, or either material. The outside cylinder *C* is of wrought iron, lined with

Air also enters the interior of the retort through the pipe *K*, which serves at the same time for discharging the product of the reaction. The excess of air and the  $\text{Cl}$  generated escape through *N*. For charging the retort, the mass is emptied into a hopper *M*, and gradually fed into the retort by a feed-screw. The fire-gases pass through *H* to the flue *P*. The process itself has undergone some alterations. Solid  $\text{MnCl}_2$ , or a solid mixture of  $\text{MnCl}_2$  and  $\text{MgCl}_2$  or  $\text{CaCl}_2$  is powdered, and mixed with powder of the solid residue from the fourth operation (see previous patent, 1882). The residual liquor from the treatment of a manganite with  $\text{HCl}$  must be neutral before it can be boiled down. A magnesium or calcium-manganite is neutralized with  $\text{MgO}$  or  $\text{CaO}$ ; a manganese manganite solution is made to run down a tower, where it meets with hot fire-gases which carry away steam and  $\text{HCl}$ , which are condensed in the usual way. In a former patent it was proposed to mix  $\text{MnCl}_2$  or a mixture of  $\text{MnCl}_2$  and  $\text{MgCl}_2$  or  $\text{CaCl}_2$ , with a portion of the products containing  $\text{MgO}$  and  $\text{MnO}$  which was already exposed to air and heat. This was done to avoid the fusing of the mass. In using the cylindrical retort, the mixing can be dispensed with, provided the furnace is heated long enough and in such a manner that the heat at one end of the retort, where the charge enters, is below the fusing point of  $\text{MnCl}_2$ , or the mixed chlorides. The mixing then takes place in the retort itself before the chlorides reach a point hot enough to fuse the mass.—S. H.

*Improvements in and Relating to the Manufacture of Chlorine.* Eng. Pat. No. 5868, Dec. 27, 1883. Walter Weldon.

This patent was granted for a method of obtaining solid chloride of manganese from the residual solutions resulting from the action of hydrochloric acid upon oxides of manganese by a process patented on August 23, 1883. The residual solutions above referred to are evaporated, and when a certain degree of concentration is reached the liquid is agitated by mechanical means, and the evaporation continued until only solid matter remains in the vessel. The chloride of manganese is thus obtained in crystals or small masses containing but little water of crystallisation. These crystals are then subject to a higher temperature, so as to drive off all the water after having been previously powdered. The evaporation may also be conducted in such a way that the crystals of chloride of manganese as they are formed can be "fished" out of the evaporating vessel. Anhydrous chloride of manganese thus obtained is heated in a current of air, whereby chlorine is driven off and oxides of manganese are left behind, which are capable of liberating strong chlorine when treated with hydrochloric acid. In order to prevent the chloride of manganese from fusing into large agglomerated masses it is mixed with oxides of manganese obtained as above, or else the heat is regulated in such a way that it shall not reach the fusing point of



fire-bricks. The whole rests and turns on rollers *D*. Pipes *F* conduct producer gas into the annular combustion-chamber *X*; openings *e* supply the necessary air.

the chloride of manganese until the chloride has been partially decomposed, and has thus become mixed with oxides of manganese.—E. J. B.

*Recovery of Sulphur from Sulphides of Alkaline Earths, e.g., Soda-Waste, by treating them with Carbonic Acid.*  
 F. B. Rawes, Stratford, Germ. Pat. 25771, 1882.  
 Dingl. Polyt. J. 253 [4] 158.

THE  $\text{CO}_2$  is taken from fire-gases, which enter through pipe A (Figs. 5 and 6) into the apparatus B, and after

Q which are arranged in such a manner that each can form alternately the first, intermediate, or last member of a series, and while one is exposed to the action of  $\text{CO}_2$ , another can be emptied and refilled without interrupting the process. The sulphide is charged through the manhole  $j$ , and rests on the false bottom  $n$ . An alkaline liquor is then made to run from

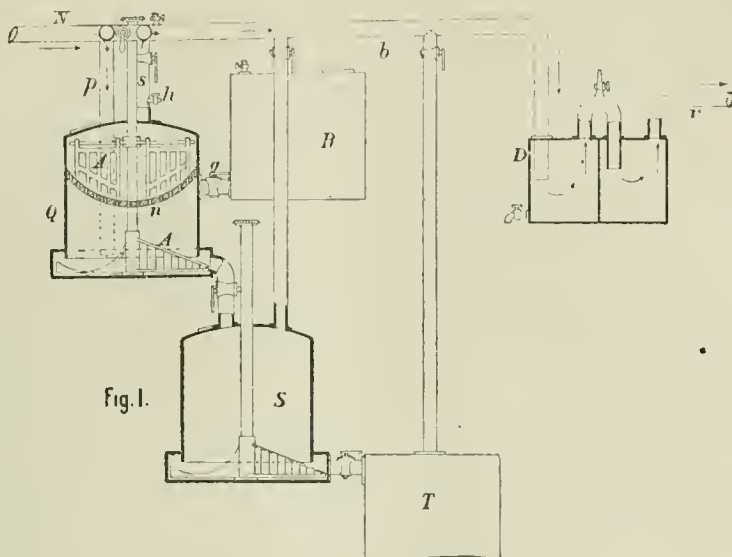


Fig. 1.

cooling are absorbed by a concentrated solution of a monocarbonate. A bicarbonate is thus formed, which on heating to  $150^\circ \text{C}$ . gives off pure  $\text{CO}_2$ . This gas enters

R into Q or P, in order to drive out the air.  $\text{CO}_2$  is now allowed to enter the cylinder, and presses a portion of the liquor in Q back to R. The connection between R

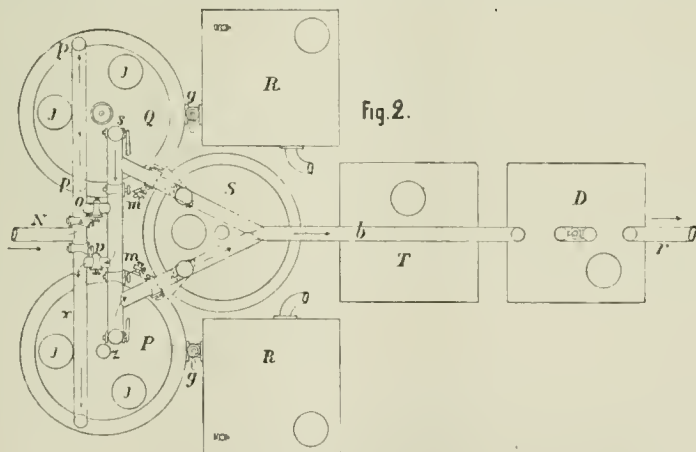


Fig. 2.

through the pipes N and p (Figs. 1 and 2) the bottom of the cylinders Q and P, which are filled with soda waste.  $\text{CO}_2$  is absorbed, and  $\text{H}_2\text{S}$  given off. The rest

and Q is then interrupted and the agitator A set to work while the action of the  $\text{CO}_2$  is continued till a sample taken at  $m$  shows that the soda-waste is desulphurised.

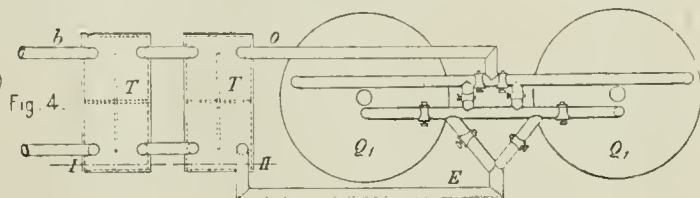
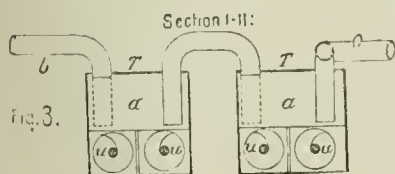


Fig. 4.

of the  $\text{CO}_2$  is retained by a monocarbonate in the absorption chamber D, and the  $\text{H}_2\text{S}$  so purified leaves at  $r$  to be reduced to S by  $\text{Fe}_2\text{O}_3$ . There are several cylinders P and

The gas is then shut off, and the contents of A run into S, where it settles. The supernatant liquor is pumped to R and used over again. The sediment is washed in T.



Tanks T (Figs. 3 and 4) divided in compartments by partition-walls serve for oxidising  $\text{FeO}$ , and reducing

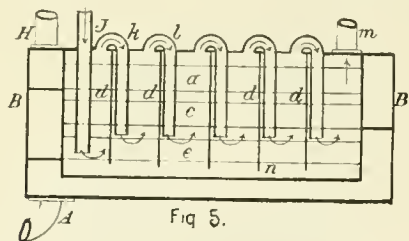


Fig. 5.

or decomposing  $\text{H}_2\text{S}$ . At the bottom there are endless screws  $u$  to produce a motion of the charge. Fire-gases enter through pipe  $b$  the tanks T, which are filled with

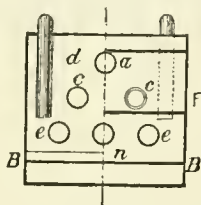


Fig. 6.

$\text{Fe}_2\text{O}_3$  and  $\text{H}_2\text{O}$  or a weak acid, and leave the last tank through pipe  $o$  to the desulphurisers  $Q_1$ , while the gas which is not absorbed in  $Q_1$  re-enters T. In the first tanks the gases give off  $\text{O}$  and oxidise  $\text{FeO}$  to  $\text{Fe}_2\text{O}_3$ . In the desulphurisers the  $\text{CO}_2$  liberates  $\text{H}_2\text{S}$ , which enters along with the other gases the second compartments of the tanks T, where it acts on  $\text{Fe}_2\text{O}_3$ , liberating S, and reducing  $\text{Fe}_2\text{O}_3$  to  $\text{FeO}$ . The screws  $u$  press the  $\text{FeO}$  into the first compartment to be oxidised, and afterwards return it to be acted upon by  $\text{H}_2\text{S}$ .—S. H.

*Improvements in the Manufacture of Bicarbonate of Soda by means of Ammonia, and in apparatus therefor, and for regenerating the Ammonia.* Eng. Pat. No. 5544, Nov. 27, 1884. La Société Anonyme des Produits Chimiques de l'est. Represented by Benjamin Didier Maguin. Communicated by Alexander Melville Clark.

THIS patent was granted for an improved arrangement whereby the absorption of the ammonia gas by brine in the ordinary ammonia soda process is made more rapid and more perfect. It also relates to an improved apparatus for the regeneration of the ammonia. The specification is accompanied by elaborate drawings, without the aid of which it is impossible to explain the apparatus.—E. J. B.

*Manufacture of Soda.* Eng. Pat. No. 5605, Dec. 3, 1883. Walter Weldon.

THIS invention consists in the manufacture of soda in such wise as to combine the advantages of what is known as the Leblanc process, with those of what is known as the ammonia process. It depends upon the reaction between sodium sulphate and ammonium bicarbonate. Hitherto the objection to such a process has been due to the fact of the comparative insolubility of sodium sulphate. Thus, if a solution of sodium sulphate were used instead of brine, for every 100 parts of water used only 11 parts of sodium sulphate could be dissolved, producing about 13 parts of bicarbonate of soda, of which 9 parts would remain dissolved in the mother-liquor, leaving only 4 parts to precipitate. This difficulty is overcome by the inventor by the use of sodium sulphate in the solid form. Several modifications of the process are given, but the following will give a general idea of the principles involved:—"I begin with a solution of ammonia and sodium sulphate, the quantity of the latter being as large as the quantity of water used will dissolve, and the quantity of the former being such as corresponds to as much ammonium sulphate as will form a saturated solution with the quantity of water used. I then treat the

mixed solution by carbonic acid gas. As the reactions that are thereby caused to take place proceed, the water present becomes capable of dissolving more sodium sulphate, and therefore I keep adding solid sodium sulphate as those reactions go on, until I have at length obtained a mother-liquor saturated with ammonium sulphate and bicarbonate of soda or ammonium, as the case may be."—E. J. B.

*Manufacture of Soda and Chlorine Compounds from Sodium Chloride.* Eng. Pat. No. 5712, Dec. 11, 1883. Eustace Carey and Ferdinand Hunter.

THIS invention relates to the manufacture of soda by a modification of the ammonia process, the modification consisting in the substitution of sodium sulphate for sodium chloride. Sodium sulphate is dissolved in water until the solution marks from  $50^\circ$  to  $60^\circ$  Tw. The solution is then freed from iron, lime, and free sulphuric acid, by the addition of a certain quantity of sodium carbonate solution. After filtering or allowing the precipitated impurities to settle, the solution is allowed to cool to a temperature of  $100^\circ$  Fah., and is then charged with ammonia to the extent of 24 or 25 parts  $\text{NH}_3$  for every 100 parts of sulphate of soda present. The solution must never be allowed to cool down below  $90^\circ$ , as in that case crystals of sodium sulphate would separate out. On the other hand it should not be allowed to rise above  $100^\circ$ , as the pressure necessary for the completion of the process would be inconveniently great. Carbonic acid is now introduced in such a quantity as would form bicarbonate of ammonia with the ammonia present. The carbonic acid should be introduced as soon as the solution is sufficiently ammoniacal, as sulphate of soda is more soluble in solutions of carbonate of ammonia than in solutions of caustic ammonia. When, in the process of carbonating, the ammonia has become converted into monocarbonate, it is necessary, in order to complete its conversion into bicarbonate, to force the carbonic acid in under pressure. The following rule is given for the calculation of the necessary pressure at  $90^\circ$  temperature: Divide the number 550 by the percentage of carbonic acid in the impure gas used for carbonating. The result, minus 15, gives the number of pounds per square inch above the atmospheric pressure at which it is necessary to work. For higher temperatures larger numbers must be used, thus, for  $100^\circ$  divide the number 950 by the percentage of carbonic acid. When sufficiently carbonated, bicarbonate of soda separates as a fine crystalline powder; the solution is then allowed to cool, so that as little as possible remains dissolved. The bicarbonate of soda is washed with water and pressed to free it as much as possible from mother-liquor; it is then heated to convert it into mono-carbonate, the carbonic acid and traces of ammonia escaping being utilised in subsequent operations. The residual liquor from the original process, containing sulphate of ammonia, bicarbonate of ammonia, and sulphate of soda, is treated in various ways to recover its ammonia. Complete drawings and descriptions of the necessary plant accompany the specification.—E. J. B.

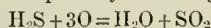
*Improvements in obtaining Sulphur from Sulphuretted Hydrogen.* 5959. 1883. C. F. Claus.

THIS and the following two specifications describe merely some additions to, and modifications of, as well as applications of, the process or processes previously patented by the same inventor, such having already been described in former numbers of this journal. The present specification describes an invention which consists in treating either pure or impure sulphuretted hydrogen for the obtaining of free sulphur therefrom, by passing it in admixture with either a little more or a little less free oxygen than the quantity necessary to form water with the hydrogen of the sulphuretted hydrogen, through layers of fragments of suitable solid materials, maintained at a suitable temperature by the heat of the reaction, or by heat obtained from extraneous sources, in order to obtain as free sulphur as much as is desired, the remainder of the sulphur of the sulphuretted hydrogen passing off

either as undecomposed sulphuretted hydrogen, or as sulphurous acid, being absorbed by any well known means.—C. F. Cl.

*Improvements in the Treatment of Mixtures of the Sulphuretted Hydrogen and other Gases, to obtain the Sulphur of their Sulphuretted Hydrogen as Sulphurous Acid.* C. F. Claus, London. Eng. Pat. 5960, 1883.

THE invention consists in a method of burning into sulphurous acid and water the sulphuretted hydrogen contained in mixtures of sulphuretted hydrogen and other gases, and especially the sulphuretted hydrogen contained in such mixtures of gases as are obtained by treating alkali waste by products of combustion, or by the gases from limekilns. So much atmospheric air is added to the gases containing the sulphuretted hydrogen, that the total quantity of free oxygen in the resulting mixture of gases shall be equal to the quantity necessary for the reaction:



The said mixture of gases is then passed through a layer of fragments of solid materials, such as oxide of iron, oxide of manganese, oxide of zinc, lime, carbonate of zinc, or carbonate of lime, or fragments of brick or asbestos coated or impregnated or not with a metal, a metallic oxide, or salt. With these substances a kiln of suitable construction is filled, and when such substances consist either wholly or in part of hydrated oxide of iron, hydrated oxide of manganese, or the like, it need not be heated up before commencing to pass into the kiln the mixture of gases containing sulphuretted hydrogen and free oxygen. Such hydrated oxides can start the reaction, but the heat developed by that reaction soon renders the said oxides anhydrous, or nearly so; but once this temperature is attained, the reaction continues notwithstanding these oxides have become dehydrated.—C. F. Cl.

*Improvements in obtaining from Alkali-Waste either Free Sulphur, or Free Sulphurous Acid, or Free Sulphur and Sulphuretted Hydrogen.* 5958. C. T. Claus, London.

THIS invention consists in a combination for the purpose of obtaining sulphur from alkali-waste, of the method of liberating the sulphur of alkali-waste as sulphuretted hydrogen, by treating alkali-waste in presence of water by carbonic acid gas (such carbonic acid gas being mixed or not with other gases), with the method of treating the resulting sulphuretted hydrogen, or mixture of sulphuretted hydrogen and other gases, by the inventor's previously described process for obtaining sulphuretted hydrogen.—C. F. Cl.

*Process and Apparatus for the Manufacture of Soda by the Ammonia Treatment.* La Société Anonyme des Produits Chimiques du Sud-Ouest. Eng. Pat. No. 7107, May 1, 1884. Communicated by John Imray.

THE apparatus consists of a series of cylindrical vessels gradually decreasing both in size and relative distance. In the centre of each a hollow perforated shaft is placed, forming the axis of an agitator; these are connected together by a branch tube, the vessels are also connected together by means of overflow pipes. An ammoniacal solution of salt is made to flow through the series commencing at the highest; meanwhile a stream of carbonic acid traverses the apparatus in an opposite direction. The crystalline bicarbonate of soda obtained is heated in a closed vessel for several hours with pure water, when it gives off ammonia and carbonic acid. Ultimately a clear solution of monocarbonate of soda remains, which may be crystallised or utilised in any way.—E. J. B.

*Improvements in obtaining from Alkali Waste Sulphuretted Hydrogen and Calcium Carbonate.* Dr. Heinrich von Miller of Krüschau, Austrian Silesia. 7847. Communicated to F. W. Renaut, Burstow, Surrey.

In patent No. 2334, 1884, the inventor described a method of obtaining a solution of sulphhydrate of calcium by treating with steam a mixture of alkali waste and water, sul-

phuretted hydrogen being injected into the mixture. He then proposed to evaporate this solution, thereby obtaining calcium hydrate and sulphuretted hydrogen. According to the present invention he decomposes the solution of sulphhydrate of calcium by carbonic acid gas, mixed or not with other gases, thereby obtaining carbonate of calcium and sulphuretted hydrogen. To obtain the sulphuretted hydrogen as free as possible from carbonic acid gas, the mixture of sulphuretted hydrogen and carbonic acid passing off from one decomposing apparatus is passed through further quantities of the solution of sulphhydrate of calcium until the whole of the carbonic acid is taken up.—C. F. Cl.

*Valuation of Commercial Potash.* Dingl. Pol. Jour. 252, p. 530 to 532.

OF late the commercial value of potash has been no longer determined on the total amount of carbonate present but only on the amount of potassium carbonate, as potash, particularly that prepared from beet sugar residues, frequently contains considerable quantities of sodium carbonate. W. F. Gintl (*Berichte öst. Chem. Ges.* 1883, 123) discusses a simplified method of investigation by which he arrives at the following method: The carbonic anhydride and potassium oxide are estimated directly; a particular sample gave 29.25 per cent. of the former and 54.42 per cent. of the latter. Now pure potassium carbonate contains 31.88 and 68.12 respectively of these, and  $68.12 \times 0.859 =$  the amount of sodium oxide per cent. in pure sodium carbonate. Now  $68.12 - 54.42 = 13.7$  per cent. of potassium oxide wanting. This  $13.7 \times 0.859 = 11.768$ , a first approximation for the sodium oxide in the sample. Next  $11.768 \times 0.1637 = 1.925$ , the corresponding increase of carbonic anhydride, added to  $31.88 - 29.25 = 2.63$ , an expression for the carbonic anhydride equivalent to the other acids present. This, multiplied by the factor  $0.2139 = 0.973$  as the increase in potassium oxide, which would have been found if all had been present as carbonate. Now we have the potassium oxide difference, e.g.,  $13.7 - 0.973 = 12.727$ , the corrected figure, and this multiplied by 0.859 gives 10.93 as the amount per cent. of sodium oxide present. The analysis of the sample was as follows:—

Water .....	0.528
Insoluble .....	0.080
Sulphuric anhydride .....	0.829
Silica .....	0.487
Chlorine .....	2.028
Phosphoric anhydride .....	1.373
Carbonic anhydride .....	29.243
Potassium oxide .....	54.428
Sodium oxide .....	11.258

The factor 0.1637 is the increase of carbonic anhydride for each unit per cent. of sodium oxide present. The factor 0.2139 is obtained as follows: The average value of the acids other than carbonic may be expressed in potassium oxide per cent. as 61.30; this subtracted from 68.12 gives 6.82, and  $6.82 : 31.88 = 0.2139$ . From the results thus obtained the quantities of potassium and sodium carbonates are obtained by finding the potassium oxide corresponding to the carbonic anhydride difference, subtracting this from the total potassium oxide found, and calculating the remainder as carbonate, whilst the sodium is calculated directly as carbonate; consequently the phosphoric acid is considered to be combined with the potassium, which is the view usually taken.—J. T.

*Decomposition of Cements by Water.* By H. Le Chatelier. *Chemisches Centralblatt*, 15, 451.

THE author has already by the optical examination of thin laminae of cements shown the presence of crystallised calcium hydrate  $\text{Ca}(\text{OH})_2$ , and crystallised calcium aluminate,  $\text{Al}_2\text{O}_3 \cdot 4\text{Ca}(\text{OH})_2$ . In continuation of these results he has studied the progressive decomposition of cements by water. Hydrated cements when treated with excess of water give up lime; it has hitherto been supposed that the dissolved lime was free lime, and it was determined in this manner, hence the varying results obtained in different laboratories. These amounts



are proportional to the water used, the calcium salts ceasing to be decomposed when the water contains a certain percentage of lime. The free lime may, however, be determined by solution by using very little water at a time, and only removing it on becoming saturated (1.3 grms. CaO per litre). In this manner no calcium compounds will be decomposed, calcium ferrite, the least stable compound of all, only beginning to decompose when the solution contains about 0.62 grms. CaO per litre. It was found in this manner that the slowly hardening cements always contain a large amount of free lime, whereas the quick setting are almost free from it. By the progressive action of water each of the constituents is decomposed in turn, giving a particular amount of lime per litre in the water, which amounts remain constant for each lime compound decomposed. By comparison with synthetically prepared bodies, the author has found the following compounds :

CaO.HO yielding a solution of 1.3 grms. lime per litre.					
Fe <sub>2</sub> O <sub>3</sub> .4CaO.12HO	"	"	0.6	"	"
Al <sub>2</sub> O <sub>3</sub> .4CaO.12HO	"	"	0.2	"	"
SiO <sub>2</sub> .CaO.3HO	"	"	0.05	"	"

The question cannot be completely answered in this manner, as in the solution of the lime there are certain stopping places, corresponding to which there have been as yet no lime compounds prepared synthetically. The author is therefore inclined to the opinion, that silico-aluminates and silico-ferrites are formed in hardening, although he has not as yet succeeded in preparing them artificially.—F. L. T.

## X.—METALLURGY, MINING, Etc.

*Preparation of Zinc free from Arsenic.* By F. Stolba. *Chemisches Centralblatt*, 15, 419.

ZINC free from arsenic and nearly free from iron is easily obtained from the commercial metal when it is simultaneously submitted to the action of sulphur vapour and steam. Burnt gypsum is mixed with about a quarter of its weight of coarsely powdered sulphur, and the mixture made into balls of about 5cm. diameter with a sufficiency of water. These balls are sunk in any convenient manner to the bottom of the crucible through the molten metal, sulphuretted hydrogen and sulphur vapour being given off abundantly and throwing the metal into active agitation. This operation is repeated if required. By treatment with sulphur alone, or steam alone, zinc can be freed from arsenic, but to remove the iron better results are obtained from their simultaneous action.—F. L. T.

*Influence of Chemical Composition on the Welding of Iron.* By Böhme. *Chemisches Centralblatt*, 15, 462.

ACCORDING to Weddell the molecular arrangement of the iron has far greater influence on the welding than the amount of carbon has. To generalise from a few analyses, the capacity of welding increases with the amount of silicon and decreases with the amount of manganese present. Reiser's idea that the capacity of welding is only influenced by the foreign substances present, in so far as they affect the crystalline structure of the iron, is more probable than Ledebur's idea, viz., that the foreign substances present have a deleterious influence on the welding property of iron proportional to their amount.—F. L. T.

*Nickel Plating of Zinc.* Ebermeyer. *Chemisches Centralblatt*, 15, 461.

THE zinc is cleaned by dilute hydrochloric acid and thoroughly washed. It is then hung in the nickel bath for a short time and on taking out is rinsed and thoroughly scraped, so removing all that does not adhere firmly. This is repeated till the zinc is covered with a thin film of nickel which can afterwards be made as thick as required. The suitable current strength is easily found. When the zinc is once thoroughly covered, the current may be increased without any risk of peeling off. F. L. T.

*Preparation of Magnesium.* By J. Walter. *Dingl. Polyt. J.*, 252, 337.

IN the *Jahrb. d. Chem. Techn.*, 1865, is contained an account of the various suggestions for the preparation of metallic magnesium. The author has investigated the probable consequence of a statement therein contained that a magnesium brass can be prepared by substituting dolomite for the zinc ore ordinarily used, viz., the possibility of preparing the metal by the reduction of the oxide by carbon, under the conditions of the zinc furnaces. Experiments were conducted at a low red and at white heat, suitable precautions being taken to isolate the metal if formed, whether distilled or volatilised. The results were entirely negative. The possibility of the formation of the metal, under the special conditions of the preparation of the alloy above named, the author sees no reason to doubt.—C. F. C.

*Progress in Metallurgy.* *Dingl. Polyt. Journ.* 252 pp. 515-519.

*Aluminium.*—H. Niewerth, Hannover (Ger. Pat. 26182, Jan. 25, 1883), mixes ferrosilicon with aluminium fluoride in equivalent proportions, and heats to fusion; volatile silicon fluoride is formed together with an alloy of aluminium and iron. On fusing this alloy with copper, a copper-aluminium alloy is said to be formed, and only a small amount of aluminium remains with the iron. If aluminium chloride be employed instead of the fluoride, silicon chloride and an aluminium-iron alloy are said to be formed.

R. F. Föhr, Schwarzenberg (Ger. Pat. 24989, April 17, 1883) proposes to extract metals from certain ores, burnt pyrites, slags, &c., in revolving barrels or similar apparatus, by means of a weak solution of magnesium chloride, calcium chloride, or sodium chloride, with or without heat. By this means the principal amount of copper, lead, &c., passes into solution. The earthy residue is then treated with bromine water for some time with exclusion of daylight; gold goes completely into solution; silver only partially so; all sulphides of copper, lead, zinc, &c., are decomposed. Copper, zinc, tin, &c., are dissolved, whilst lead sulphate remains behind. Finally the ore is treated either with the solution first obtained or a fresh chloride solution is employed, whereby silver and lead are completely dissolved. The solutions obtained are treated separately, or together with manganese dioxide and sulphuric acid, to recover the bromine, and the heavy metals are precipitated by means of iron, sulphuretted hydrogen, or other known means.

C. A. Hering, Bischofshofen, Austria (Ger. Pat. 26101, Aug. 19, 1883) proposes to obtain antimony from poor ores and other lixiviation residues, by subliming in a gas reverberatory furnace heated to a temperature sufficient to volatilise the antimony. Antimonious and antimonious oxides are produced, and are easily condensed in suitable apparatus. If arsenic be present it is also sublimed, and is found in the more remote portions of the condensing apparatus. The oxides can be fused to regulus, and the raw regulus refined in a reverberatory furnace to *antimonii regulus stellatus* of complete purity.

Tungsten steel (I) and tungsten iron (II) have, according to L. Schneider and F. Lipp, the following composition:—

	I.	II.
Iron .....	85.000	68.363
Tungsten .....	11.028	28.181
Manganese .....	1.493	0.986
Cobalt and nickel .....	traces	traces
Silicon .....	0.263	0.233
Phosphorus .....	0.007	0.008
Sulphur .....	traces	traces
Carbon .....	2.117	1.882
	99.938	99.653

The analysis is made by covering the sample with water, then adding gradually twice the amount of bromine; after warming gently nitric acid is added, and the whole is taken to dryness on the water-bath; this process is repeated; finally the mass is taken up with dilute nitric acid. The undissolved mass is fused with sodium carbonate to

separate the last portion of iron oxide, extracted with water, and the solution is evaporated to dryness with nitric acid, and the mixed silica and tungstic oxide weighed. These are best separated by fusing with five times their weight of acid potassium sulphate, and extracting with a dilute solution of ammonium carbonate; the whole of the tungsten oxide is dissolved and silica remains behind. The estimation of carbon in tungsten steel is made without difficulty by the copper chloride process; but the solution has no action on tungsten iron, so that the estimation can only be made by a direct combustion in oxygen.—J. T.

*The Formation and Working up of Slags.* Dingl. Polyt. Journ. 253 [4] and [5], pp. 163-173 and 204-206.

M. WEBER has published the following analyses of Scotch blast-furnace slag:—

	Granular.	Vitreous.	Old System.
SiO <sub>2</sub> .....	57.95	52.71	43.24
Al <sub>2</sub> O <sub>3</sub> .....	21.96	21.44	29.93
CaO .....	16.24	19.13	25.18
MgO .....	.43	3.70	.88
Ca .....	1.67	1.12	—
S .....	1.33	.90	—
MnO .....	.37	.64	—
FeO .....	.05	.36	—
	100.00	100.00	99.23

At the general meeting (1883) of the German Iron-masters, G. Hilgenstock read an exhaustive essay on the behaviour of phosphorus in the blast-furnace. He especially investigated the question as to whether all the phosphoric acid introduced into the furnace along with the charge is found as phosphorus in the pig-iron, or whether the gases of the furnace-throat contain volatile phosphorous compounds. His results were as follows:—

	I.	II.	III.	IV.
Brought into the furnace along with the slag (calculated on 100 Fe) ....	3.235	3.235	3.25	3.25
Less P in the slag .....	.24	.07	.33	.225
Leaving .....	2.995	3.165	2.92	3.025
Actually found in the pig-iron .....	2.76	2.74	3.18	3.41

The difference is, of course, explained by the extreme difficulty of obtaining a fair average sample of the pig-iron and the slag. The charge contains the phosphoric acid as iron or calcium phosphate, and as iron has a very great affinity for phosphorus, phosphoric acid is reduced to phosphorus, which combines at once with iron and no free phosphorus can be present. The latter cannot volatilise either, since at a red heat it burns to phosphoric acid in the presence of carbonic acid. This reasoning is confirmed by the fact that no trace of phosphorus could be detected in the gases issuing from the furnace top. The following analyses show that the percentage of phosphorus in the slag rises with the increase of phosphorus in the charge. They also prove that the less the quantity of the reducing agents present, and the lower the temperature, the more phosphoric acid will be found in the slag as unredacted P<sub>2</sub>O<sub>5</sub>:—

	Pig-Iron.				Slag.	
No.	Si	P	Mn	C	P	
1. ....	trace	5.96	0.92	0.88	2.57	
2. ....	trace	7.20	0.36	1.11	2.39	
3. ....	0.02	6.24	0.51	0.95	1.74	
4. ....	0.06	6.07	0.75	1.19	1.22	
5. ....	0.09	4.57	1.98	0.90	0.38	Charge
6. ....	0.28	3.61	1.69	1.19	0.18	less
7. ....	0.28	3.79	1.13	1.12	0.19	phosphatic.

Considering that the furnace was working during these experiments with a rather high proportion of coke, the small amount of Si and C is surprising. Direct experiments proved that these elements diminish with an increase of phosphorus in the iron.

3.26% P .....	1.03% Si .....	2.01% C
12.12% P .....	0.02% Si .....	0.87% C

The rising and falling of the Mn curve in the above-mentioned analyses is due to the rise and fall of the temperature, and with more heat a higher percentage of P and Si can also be attained. Iron alloys with phosphorus just like manganese, but the fact is remarkable that with a high percentage of P iron ceases to be magnetic. 9.46% P does not alter its magnetic

properties; iron with 16% P was but weakly attracted by a powerful magnet, whereas iron with 25.6% P was not attracted at all. The analysis of irons containing much phosphorus is extremely difficult. Such iron dissolves only slowly in dilute nitric or hydrochloric acid without leaving any perceptible residue. For determining carbon, the author did not succeed in dissolving it in cupric-ammonium chloride, or by treating it with iodine and water at 0° C. The iron must therefore be volatilised by heating it in an atmosphere of chlorine, and the carbon can then be estimated by burning it in oxygen, etc. Numerous experiments have been tried with a view of preventing phosphorus from combining with iron. As it was well known that phosphoric acid is reduced by carbon, water-gas, i.e., CO and H<sub>2</sub>, was suggested for the reduction of iron ores. But these gases, while they reduce FeO and Fe<sub>2</sub>O<sub>3</sub>, also act upon phosphoric acid. Tribasic iron phosphate is reduced by H at a bright red heat, and, according to Finkener, CO reduces that compound in the presence of considerable quantities of iron peroxide. Therefore phosphoric ores can never be expected to yield iron free from phosphorus by the reducing action of CO and H<sub>2</sub>, even if these gases are used in a pure state. All processes aiming at the direct production of iron and steel, and based on this principle, must be accepted with the greatest reserve. A. Ledebur (Stahl und Eisen, 1884, 249) holds that liquid slag is a solution of different oxygen compounds, whose components may group themselves in different manners according to the temperature at which they solidify. A slag from the Martin process poured into a small iron mould had a vitreous appearance and olive-green colour (I), where it cooled quickly, whereas a portion which cooled more slowly was opaque (II). The analysis showed:—

	I.	II.
SiO <sub>2</sub> .....	48.03	48.10
Al <sub>2</sub> O <sub>3</sub> .....	1.60	1.85
FeO .....	16.23	16.66
MnO .....	31.53	31.67
CaO .....	—	1.08

The small differences in the analyses do not warrant a safe conclusion. The slag of a charcoal blast furnace, which was allowed to cool quickly on the outside and slowly in the inside, had a vitreous appearance and olive-green colour (I), whereas the core was of a crystalline texture and ultramarine-blue (II). The analysis showed:—

	I.	II.
SiO <sub>2</sub> .....	43.43	43.43
Al <sub>2</sub> O <sub>3</sub> .....	17.80	17.28
FeO .....	1.05	1.64
MnO .....	2.09	2.41
CaO .....	33.30	31.57
MgO .....	—	trace
S .....	—	—
Alkalis and loss	2.33	0.77

Here the difference in the percentage of iron, lime and alkalis is remarkable. It is also worth mentioning that whereas the quickly cooled portion was readily decomposed by HCl, the inside part resisted even a long-continued action of the acid. The chemical composition of slags depends upon different circumstances. If, for example, the pig-iron is rich in S, Si and Mn, and provided the temperature of the blast-furnace was high, then the slag will be poor in Fe. Or if iron poor in carbon is required, the slag must be richer in Fe than in pig-iron making. Again, manganese ores yield slag poor in Fe and so forth. The formation and composition of the slag in the old refining process (the "Frischprozess" of the Germans) chiefly depends upon the temperature, the composition of the iron at the time when the sample of the slag was taken, and the compounds which go to form the slag, including the lining of the furnace. A slag containing FeO and MnO acts more powerfully on a lining rich in silica than a slag containing little or no MnO. In a furnace with a basic lining the slag contains little SiO<sub>2</sub>. In the puddling process the slag is at first rich in Fe, then decreases in Fe and remains constant till nearly the end of the process, and eventually the percentage of iron in the slag rises again as soon as most of the carbon in the iron has disappeared. The slag contains iron as FeO and Fe<sub>2</sub>O<sub>3</sub>. In the Bessemer process the percentage of iron



in the slag is always less than that of slag from the puddling process, and all the more so the higher the temperature is and the more manganese is present in the pig-iron used. After the oxidation of most of the carbon the iron becomes oxidised, and the slag thus grows again richer in iron. At the end of the process, when the temperature is very high, the amount of slag formed increases, but the percentage of iron in it diminishes. In the Martin process a high temperature and a lining of silica cause the formation of a slag rich in  $\text{SiO}_2$  and poor in Fe. In this process the iron usually contains less manganese than that worked in a Bessemer converter, and so the slag contains less manganese than Bessemer slag. The following table shows the different stages of two "Martin" operations at the rolling mills of Graz:—

centage of  $\text{SiO}_2$  in the slag is not very high, and it remains so until the final charge is brought into the furnace. When the carbon of the iron becomes less, some iron is oxidised and goes into the slag, decreasing the percentage of  $\text{MnO}$ . With the rise of the temperature, the percentage of  $\text{SiO}_2$  increases and attains its maximum at the end of the process. In the second experiment the composition of the slag is somewhat different. The first charge being larger, more heat is absorbed, and the percentage of manganese in the slag rises, and that of iron decreases. With the second charge (tyres and rails) much Mn comes into the furnace and acts in dissolving silica, and raising its percentage. But owing to the low temperature, and the richness of the slag in manganese, the oxidation of carbon in the iron proceeds more slowly than in the first experiment.

CHARGE.	IRON.			SLAG.					
	C	Mn	Si	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	MnO	FeO	CaO	MgO
<b>I.</b>									
6.40 a.m.—1st charge, consisting of— 2100 kg. Vordernberg pig-iron (white), 1500 kg. Loelling pig-iron (grey), 1000 kg. old steel.									
Sample after fusing.....	1.13	.11	.01	42.56	1.46	28.39	27.47	trace	—
9.10 a.m.—2nd charge, consisting of— 500 kg. tyres, 500 kg. iron turnings, 2000 kg. old boiler-plates, 1000 kg. old rails.									
Sample after fusing second charge	.69	.11	—	42.91	1.53	22.23	31.17	—	—
11.20 a.m.—3rd charge, consisting of— 3900 kg. old rails.									
Sample .....	.27	.13	—	48.03	1.76	18.48	30.15	.78	—
12.28 p.m.—Sample .....	.20	.12	—	47.87	2.34	19.53	29.99	—	—
1.10 p.m.—Sample.....	.12	.08	—	48.90	2.01	19.37	28.88	—	—
1.45 p.m.—Addition of 120 kg. Silicon-Ferro-Manganese.									
Average sample of the finished iron .....	.31	.15	.01	49.63	—	20.89	25.42	—	—
<b>II.</b>									
6.15 a.m.—1st charge, consisting of— 2100 kg. Vordernberg pig-iron (white), 1300 kg. Loelling pig-iron (grey), 1200 kg. old steel, 1000 kg. tyres.									
Sample after fusing.....	1.16	.21	.01	42.13	1.57	35.19	20.37	.70	—
9.0 a.m.—2nd charge, consisting of— 1500 kg. tyres, 500 kg. turnings, 1900 kg. rails.									
Sample after fusing second charge	1.10	.16	—	49.56	1.96	32.25	14.41	—	—
12.50 p.m.—3rd charge, consisting of— 3000 kg. rails.									
Sample .....	.62	.15	—	50.06	1.81	28.92	18.14	—	—
2.0 p.m.—Sample .....	.52	.11	—	51.17	1.51	29.39	17.06	.53	—
Addition of 100 kg. red hematite after wards									
1.35 p.m.—Sample.....	.19	.11	—	57.13	2.66	18.29	17.28	3.01	.60
4.15 p.m.—Addition of 120 kg. Silicon-Ferro-Manganese.									
Average sample of finished iron .....	.37	.10	.02	53.07	1.85	19.99	14.68	3.18	.11

At the commencement the temperature of a Martin | The addition of red hematite causes iron to go into the  
furnace is proportionally low, and consequently the per- | slag, diminishing the percentage of manganese. There

is considerably more  $\text{SiO}_2$  in the slag of Experiment II., probably owing to a high temperature of the furnace. The percentage of Fe, however, is lower, partly owing to the last-mentioned circumstance, but also in consequence of the presence of large quantities of manganese and a higher percentage of carbon in the iron. In all cases the percentage of  $\text{SiO}_2$  rises with the temperature and the quantity of manganese present, whereas that of iron decreases under these circumstances. The following results of the analyses of slags at the end of four Martin operations confirm this statement:—

IRON.			SLAG.			
Sample.	C.	Mn.	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	MnO.	FeO.
I.	·13	·20	50·13	1·86	17·99	29·55
II.	·22	·36	45·75	3·03	13·67	36·46
III.	·13	·17	47·26	2·06	6·58	40·11
IV.	·10	trace	50·05	4·11	7·81	35·66

At No. IV. the slag does not contain much manganese, but the high temperature of the furnace caused the slag to take up much silica. L. Garnier, of Alais, is said to have successfully used blast furnace slag as a remedy against the phylloxera in vineyards. The action of the slag is attributed to the sulphur it contains. R. Hasenclever (*Zeitschrift d. Vereins deutscher Ingenieure* 1884, 206) describes the plant at Schalke and Stolberg for converting slag of the basic process into phosphate, according to Scheibler's patent. The slag is roasted in a reverberatory furnace, with an inclined double bed, 28ft. long by 5ft. wide. Each furnace can roast from 15 to 17½ tons in 24 hours, requiring from 2 to 2½ cwt. coal (according to quality) per ton of slag. The roasted product is treated with steam, whereby the CaO contained in the slag is changed in  $\text{Ca}(\text{OH})_2$ , breaking up the stuff into fine powder. It is then sieved, to separate big lumps and pieces of iron and steel. The caustic lime is removed by elutriation, and the slag then treated with hydrochloric acid. The use of sulphuric acid has the disadvantage of contaminating the residue with sulphate of lime, and thus making it unfit for metallurgical purposes. Besides, the gypsum in surrounding the fine slag-dust prevents the complete extraction of phosphoric acid. The quantity of acid must be such as to dissolve only those earthy bases which are present in a free state, or combined with silicic and phosphoric acid. The degree of dilution is 1 vol. of 21° B. (33·5° Tw.) to 10 to 15 vol. of water, whereby a precipitation of gelatinous silica is avoided. The quantities of acid used are variable, and are larger the more free lime is present, and the greater the amount of lime combined with silicic and phosphoric acid. Thomas'-slag, made in Germany, requires 1½ to 1½ litre HCl per 1kg. slag. The action of HCl takes place in tanks provided with agitators, and is finished in a few minutes. The solution is allowed to settle, and the supernatant liquor then precipitated by milk of lime, taking care to precipitate the  $\text{SiO}_2$ , together with  $\text{P}_2\text{O}_5$ , or to keep most of the  $\text{SiO}_2$  in solution by an incomplete neutralisation of the phosphatic earths. The sediment is filter-pressed, washed, dried, and sold as calcium-biphosphate for agricultural purposes. The residue from the treatment with hydrochloric acid has the following composition:—

$\text{SiO}_2$	1·48%	3·60%
$\text{P}_2\text{O}_5$	3·00	·60
$\text{Fe}_2\text{O}_3$	49·80	68·28
$\text{Mn}_2\text{O}_3$	17·06	12·70
CaO	15·60	4·00
MgO	12·50	11·35
	99·41	100·53

It is easy to obtain by this process a product containing 35% to 37%  $\text{P}_2\text{O}_5$  as calcium biphosphate. By igniting it the percentage of  $\text{P}_2\text{O}_5$  rises to more than 45%. It is especially suitable for making superphosphate on account of its small percentage of lime, since it would require only

half as much sulphuric acid as native phosphates. The extensive working of this process must have a considerable influence on the consumption of hydrochloric acid, as 1 ton of slag requires from 1 to 1½ tons HCl. B. Platz (Stahl and Eisen, 1884, 262) has found in the hoshes and hearth of a blast furnace at Duisburg-Hochfeld strong crops of dense masses of slag, two feet in thickness, which were of a greyish black colour. Placed in water, they gave a dark red solution of potassium ferrate. In the pores there were small needle-shaped crystals, which could be crushed with the finger to a dark red mass. The masses were penetrated with a white crust of carbonate of potash. Some pieces contained bluish-green crusts of potassium manganate. Platz thinks that these deposits were separated by a dense layer of coke from the moving stream of fused slag and the reducing gases, and owing to that circumstance secondary processes could go on which are in direct opposition to the normal process of the blast furnace. Protected from the reducing gases which could not penetrate into the interior, the ferrate was formed by the action of an excess of carbonate of potash upon iron peroxide. Two samples of the dense masses, originally containing potassium ferrate, tested—

$\text{Fe}_2\text{O}_3$	13·72	7·83
FeO	21·75	12·77
MnO	·46	·92
$\text{SiO}_2$	11·98	6·61
$\text{Al}_2\text{O}_3$	6·72	3·10
CaO	33·02	59·62
MgO	1·27	1·41
CaS	1·35	1·37
	93·27	93·66

The rest consists of alkalis, chiefly carbonates and sulphates. The masses had the characteristic appearance of slag having previously been fused, and the low percentage of  $\text{SiO}_2$  leads to the belief that iron peroxide and alumina must have played the part of acids. It is also worth adding that these deposits contained enclosed well-shaped transparent crystals of anhydrous potassium sulphate (Glaserite).—S. H.

*Utilisation of Slag.* A. Frank, Charlottenburg. Dingl. Pol. Journ. 252, p. 528. Ger. Pat. 27106, September 16, 1883.

RECOMMENDS the application of magnesium chloride for the decomposition of slags containing sulphur and phosphorus. The fluid slag is run into a solution of about 1·06 sp.gr. and agitated; the sulphides are decomposed with the evolution of hydrogen sulphide; so in basic slags uncombined lime produces calcium chloride and magnesia, which indirectly induces a concentration and more easy solubility of phosphates present. The magnesia thus produced can be removed by washing and settling. On heating in an oxidising flame the slag powder obtained with the still adhering magnesium chloride, or with a further addition of chloride, a partial higher oxidation of the ferrous oxide and similar compounds results, the new compounds formed being less prejudicial to the manure obtained. Instead of beginning with fluid slag, solid slag finely ground can be heated with the magnesium chloride solution under high pressure. Similarly other phosphates can be thus treated, especially such as contain lime not combined with phosphorus, and not convertible into caustic lime by ignition, e.g., marl containing phosphoric acid. Slags and phosphates so treated can be at once applied as manure. It is, however, better to treat the phosphate compounds with magnesium chloride with the addition of ammonium chloride and hydrochloric acid, say to 1 equivalent tribasic calcium phosphate add about 1 equivalent hydrochloric acid, 2 equivalents magnesium chloride, and 1 equivalent ammonium chloride. Calcium chloride and ammonium-magnesium phosphate are formed in solution. The latter can be precipitated by neutralising with calcium or magnesium carbonate and adding caustic alkali, or alkaline earth. In place of magnesium and ammonium chlorides, the sulphates can be used along with free hydrochloric acid.—J. T.



*Modification of Roquer's Process for Working up Phosphorised Slags.* Dingl. Polyt. Journ. 253 [3], 135.

THE slag is smelted in a cupola, whereby a matt is obtained containing 20 to 25 per cent. of phosphorus. It is then mixed with powdered anhydrous  $\text{SO}_4\text{Na}_2$ , and heated to redness. Most of the phosphorus is changed into sodium phosphate, whereas a portion of Fe and Mn is converted into phosphates, sulphides and oxides. The mass is treated with water to recover sodium phosphate by crystallisation. The insoluble residue is mixed with  $\text{Na}_2\text{SO}_4$  and charcoal, and heated in a reducing flame. The  $\text{Na}_2\text{SO}_4$  is first converted into  $\text{Na}_2\text{S}$ , and then by double decomposition sodium phosphate and FeS and MnS are formed. The mass thus yields another crop of sodium phosphate crystals. The residue, after roasting to destroy the sulphides, can be used as an iron ore rich in Mn. The sodium phosphate is employed for artificial manure. Another method to work the phosphorised matt is to fuse it in a Bessemer converter with dolomite or lime. Alkali can be added to promote the fusing of the metal slag which is formed. Before the complete dephosphorisation the slag is decanted, and a fresh portion of lime added to obtain the dephosphorisation according to the basic process. The slag contains  $\text{P}_2\text{O}_5$  and only little Fe and Mn. It is powdered and used either directly as manure or after treating with  $\text{SO}_4\text{H}_2$  as superphosphate. The second method yields the P as a product of less marketable value, but as the metal has been converted into steel, its value is said to make up the difference.—S. H.

*A New Method of Working up Sheet Tin.* Ed. Donath, Löben. Dingl. Polyt. Journ. 253 [5], 206.

SHEET-TIN clippings are boiled with a concentrated caustic soda liquor and  $\text{MnO}_2$  until the mass assumes a pasty consistency. Water is then added, and the whole stirred up and allowed to settle. The decanted solution is mixed with acetic acid till it has an acid reaction,

with  $\text{H}_2\text{O}$ , yellow prussiate solution is added, which produces a white precipitate. It is boiled in the acid liquor for some time, filtered, washed, and dried on shallow dishes at a pretty high temperature, when it will turn blue. The colour compares well with the best brands in the market.—S. H.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*Determination of Glycerine in Aqueous Solution by means of the Index of Refraction.* By F. Strohmmer. Chemisches Centralblatt 15, 397.

THE author deduces from his experiments by the method of least squares, the formula

$$n_{(D)} = 75.875 + 0.56569d$$

$$\text{and since } d = \frac{(K + 100)D}{D(100 - C) + C}$$

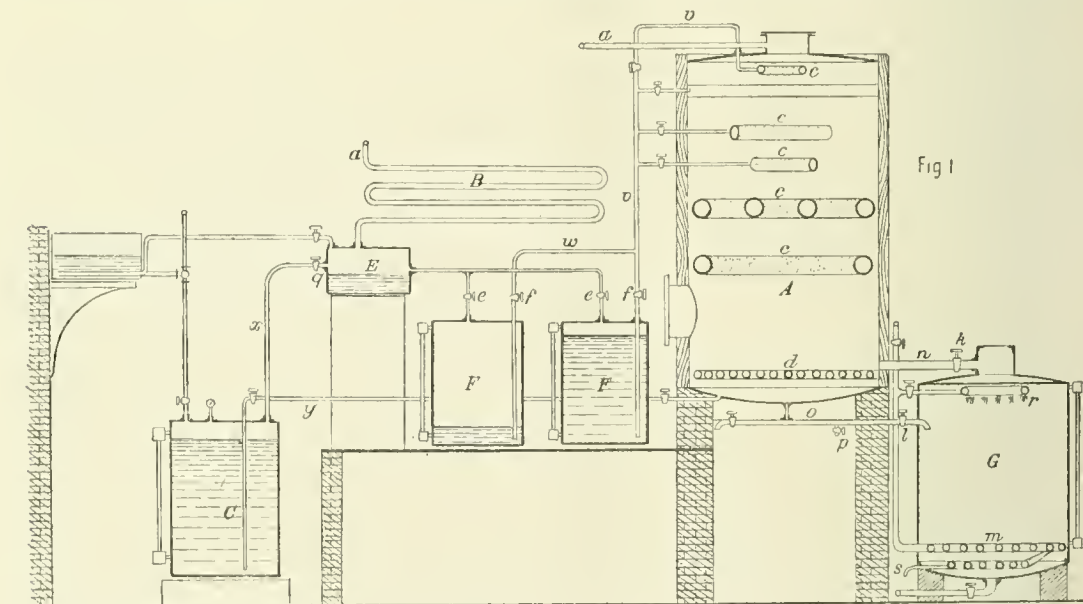
$$n_{(D)} = 0.75875 + \frac{(K + 56.569)D}{D(100 - C) + C}$$

where  $d$  = sp. g. of solution,  $D$  = sp. g. of anhydrous glycerine, which is 1.262 at  $17.5^\circ$ , and  $C$  = percentage of glycerine by weight. The rest of the paper is a claim of priority for a paper annexed, published in 1878.

—F. L. T.

*Apparatus for the Extraction of Fat from Bones.* Dingl. Polyt. Journ. 253 [3], 123.

W. SCHNEIDER, Lehrberg (Germ. Pat. No. 26687, 1883), has patented the apparatus represented in Fig. 1. The extractor A is filled with bones, and the solvent pressed from C through the pipe  $y$  into A, which is heated by a



raised to the boil, when all tin is precipitated as oxide. The filtrate is used for acetate of soda manufacture. The oxide can be converted into the different salts. The clippings can also be used for the manufacture of a Prussian blue of excellent quality. A tin salt gives, with yellow prussiate, a white precipitate, which, when washed with hot water or dried at a high temperature, turns blue, giving off  $\text{HCN}$ . Owing to this reaction, the clippings are boiled with dilute  $\text{HCl}$ , and the clear solution is oxidised with  $\text{NO}_2\text{H}$ . After strongly diluting

steam-coil  $d$ . As soon as the solvent is on the boil, the steam is shut off, and the taps  $k$  and  $l$  are opened. The solvent remaining in A flows together with the dissolved fat into G, which is then heated by steam. The solvent distils off, and the vapours passing through  $n$  back into A come here in close contact with the bones. Any escaping vapours are condensed in the cooling worm B, whence the liquor flows into storing vessels F, whence the liquor is again forced into A, and returns to the cycle of the process. From time to time samples are

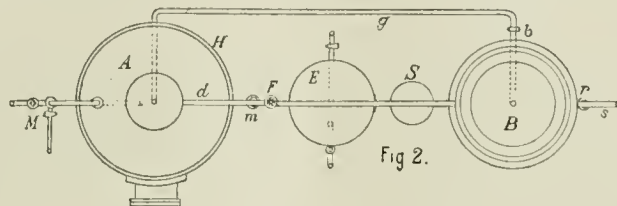
drawn from the tap *p*, and when the solvent contains only very little fat, the taps *l* and *f* are shut off, while the steam in *G* is allowed to go on for some time longer to drive all solvent into *A*. A rose *r* blows steam from the top into *G*, and prevents the boiling fat from rising and frothing over. Steam also enters *G* at *s* in order to purify the fat. All the steam of *G* escapes at *n* into *A*, where it serves to drive the solvent into the cooling worm *B*. If necessary, the temperature of *A* is raised by direct steam. For the extraction of fat and glue from bones, H. Neumeyer, Nuremberg (Germ. Pat. 26882, 1883), puts the bones in a broken state into the extractor *A* (Figs. 2 and 3), which is provided with a perforated

enough, the liquor from the bottom is repeatedly pumped to the top till it has reached the required concentration. The solution is then forced to cooling vessels through the pipe *Z*.—S. H.

## XII.—PAINTS, VARNISHES, AND RESINS.

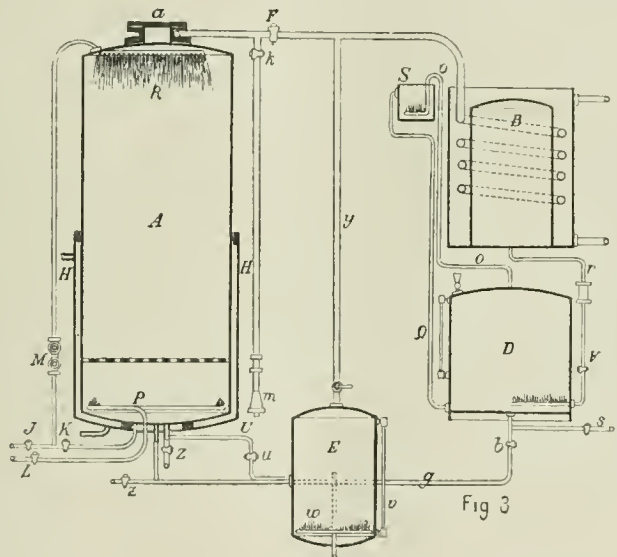
*Colouring of Amber.* By E. Hanausek. *Chemisches Centralblatt* 15, 461.

To colour amber it is necessary to heat the amber in some fluid to about 150° or 200°, which does not act upon



bottom above the real bottom. After screwing down the lid, and closing all taps except *F* and *K*, the vessels *A* and *B* are exhausted by means of an air pump or an injector. When the vacuum meter shows a certain

it, and in which the colouring matter will dissolve. Many fluids fulfil these conditions, more especially linseed oil. Among the colouring matters which dissolve in this oil, and are not decomposed at 150°–200°, are



vacuum, *k* is shut and *b* opened, whereby the solvent is sucked from *D* through the pipe *g* into *A*. The taps *b* and *f* are now closed, and steam is allowed to enter the steam-chest *H*, till a certain pressure is reached in *A*. *F* and *V* are then opened, and the vapours of the solvent pass to the cooling worm *B*, are there condensed and run back to *D*. The small amount of the solvent which adheres to the bones in *A* is driven to the condenser *B* by means of a steam rose *P*. The fat produced in *A* runs through the pipe *U* into the vessel *E*, is there purified by steam, and can be drawn off at pleasure. After finishing the extraction of the fat, the taps *F* and *V* are closed, and warm water is allowed to run over the bones from a rose *R* at the top, till it runs away clear at *Z*. A certain amount of oxalic acid or *HCl* is then forced into *A* from *M*, and acts on the bones. The acid which collects at the bottom is repeatedly pumped to the top, and divided into fine spray by the rose *R*. After a certain time the maceration is complete. The tap *K* is then closed, and steam let into *A* from *L* till there is a pressure of 45lbs. in the extractor. After standing for some time, a certain amount of hot water is run into *A* from the top. Should the solution of glue so formed not be strong

dragon's blood, alizarin, purpurin, and indigo. Neither fuchsine, aniline violet, methyl green, nor alkali blue dissolve in linseed oil. The oil and colouring matter are mixed in weighed quantities, the amber hung in the mixture, and the whole heated to 190° to 200°, kept at the temperature of 180° to 200° for a few minutes, and then allowed to cool slowly to the temperature of the air. By varying the amounts of colour used, different tints are produced; e.g., indigo gives a lighter or darker green, dark blue or black according to quantity used. To obtain pure colours the bath should be changed or renewed from time to time, except for black, when it is not so necessary.—F. L. T.

## XIV.—AGRICULTURE, MANURES, Etc.

*On the Decomposition of Nitrogenous Manure.* Dingl. Polyt. Journ. 253 [4], 175.

NITROGENOUS organic matter loses free *N* by putrefaction. An addition of gypsum usually, but not always, reduces the loss; soil is of no value, and in one case even increased the loss fourfold. The quantity of *NH*<sub>3</sub>



formed is in direct proportion to the loss of N. 10% kainite altogether arrested the loss of N at the putrefaction of horn-powder. In all cases where N was given off, the putrefying mass had an alkaline or neutral reaction; whereas in those cases where no perceptible loss of N had taken place, the reaction was slightly acid. Dietzel has observed that free  $N_2O_3$  is formed at the putrefaction, and explains the reduction to N by the well-known reaction between  $N_2O_3$  and  $NH_3$  or Amides. If this statement is confirmed, the  $N_2O_3$  can only be formed by oxidation, and the loss of N must increase with the porosity and moisture of the mass.—S. H.

*Is Cellulose a Food?* By H. Weiske. *Chemisches Centralblatt* 15, 385.

SINCE Haubner, Henneberg, and Stohmann have proved that an appreciable amount of the cellulose consumed by the cow does not reappear in the faeces, and as the same fact has been confirmed in the case of other herbivora, and for the pig, it has been concluded that the amount of cellulose that disappeared or was digested by an animal, was digested as a food like starch. Hoppe-Seyler, Popoff, and Tappeiner having shown that cellulose under the influence of specific ferments readily splits up into gaseous bodies, among which marsh gas predominates, the author in consequence of his own and the above experiments and those of Ellenberger and V. Hofmeister considers it is still questionable whether the "digested" cellulose is a food in the accepted sense of the word. In one experiment a sheep was fed daily for two weeks with 500grms. bruised beans; the sheep excreted on an average 20.93grms. nitrogen in its urine. It was then fed on a daily ration of 490grms. bruised beans and 515grms. oat straw, the digestion-coefficient of which was known. The urine now contained a daily average of 16.82grms. of Nitrogen. The animal now for a long time received daily 500grms. bruised beans and 200grms. starch, an amount of food containing the same quantity of digestible nitrogenous and non-nitrogenous food (including digestible cellulose) as the former dietary. The daily evacuation of nitrogen in the urine now sank to 14.94grms. on the average. On again dieting it on 490grms. bruised beans and 515grms. oat straw, the evacuated nitrogen averaged 17.26grms., although equal amounts of nutriment had been consumed in the starch and in the oat straw diets. It must accordingly be accepted that the nutritive substances of the oat straw, consisting of digestible cellulose and digestible non-nitrogenous extractives, have not the same nutritive value as starch; the "digestible" cellulose may perhaps have been entirely worthless. Accordingly the sheep was fed daily on 500grms. bruised beans, and only 100grms. starch (*i.e.*, just as much starch as the 515grms. of oat straw contained of non-nitrogenous extractives). The evacuation of nitrogen now rose to a daily average of 17.75grms., about the same amount as on a diet of 490grms. bruised beans and 515grms. oat straw. In complete agreement with these results are the daily amounts of nitrogen taken up or given off by the animal (*i.e.*, the differences between the total amounts of nitrogen taken up in the form of food and the amounts lost in urine and faeces). With a diet of bruised beans entirely, there was a loss of 0.34grms. Diet of bruised beans and oat straw, a gain of 2.94grms. Diet of bruised beans and 200grms. starch, a gain of 5.06grms. Diet of bruised beans and 100grms. of starch, gain of 2.96grms. The author concludes, notwithstanding all previous suppositions, that cellulose has no saving influence on albuminous bodies similar to that exerted by starch and other digestible carbohydrates and the fats.—F. L. T.

*Silica as Manure.* *Dingl. Polyt. Journ.* 253, 174.

C. KREUZHAGE and E. WOLFF have inquired into the value of  $SiO_2$  for the cultivation of oats, and whether the assimilation of  $SiO_2$  from the soil is followed by a better yield of the common agricultural plants. They found that the formation of the grain is more complete if the plant can take up  $SiO_2$ . Previous experiments prove that the increase of  $P_2O_5$  in the liquid of the soil acts very

favourably on the quality and quantity of the grain produced. But the  $SiO_2$  cannot replace  $P_2O_5$  in the manure, and we have yet to learn in what form the  $SiO_2$  is best assimilated by the soil. The experiment to bring so-called "Silica-Poudre" into the market was a complete failure.—S. H.

## XV.—SUGARS, GUMS, STARCHES, Etc.

*Preparation of Maltose.* By L. Cuisinier, *Chemisches Centralblatt*, 15, 413.

THE author describes Dubrunfaut's method of obtaining maltose with the modifications necessary on the large scale. The maltose can be prepared either in crystals or as syrup.

1. *The Water* must be free from suspended impurities and organic matter, and should contain neither carbonate nor sulphate of lime, the former promoting butyric fermentation, and the latter interfering on evaporation. Distilled water is accordingly recommended.

2. *Raw Material.* For crystals—starch in the purest condition; for syrup—meal, potatoes, grain, the latter being coarsely ground.

3. *Preparation of the Malt.* In the manufacture of crystallised maltose, malt itself is not used, because some of the little known substances which it contains have a deleterious influence on the crystallisation. An aqueous infusion made at 30° is accordingly employed. The malt must be dried at a low temperature, or green malt may be used. Instead of barley other malted grains may be used.

4. *Solution of the Starch.* The starch is made into a paste with twice its weight of water, and, after an addition of an amount of extract equal to 5 per cent. of malt, is thoroughly mixed. An amount of water, equal to ten times the weight of starch, is heated to 90°, and the two liquids allowed to flow simultaneously through a peculiar sieve. The solution is ended by blowing in steam. Temperature of the mixture on entering the boiler 75°. The mass is as mobile as water at 90°, and the operation may be considered at an end.

5. *Saccharification.* The fluid is conveniently cooled to 40° and treated with an amount of malt extract equivalent to 10 to 15 per cent. of malt on the original starch. The temperature during saccharification is maintained between 40° and 50°. The progress is ascertained by means of iodine solution, and in the preparation of crystalline sugar also by the alkaline copper solution. After 2 or 3 hours the fluid no longer gives a colour with iodine, and in the manufacture of syrup the process may be stopped; if solid maltose is required the operation must be continued for 12 to 15 hours.

6. *Filtration and Concentration of the Liquor.* When only starch is employed filtration is sufficient to separate the fluid from the residue, but when rice, maize, &c., are used a preliminary pressing is necessary. The fluid, passed through an Oldham-Farquhar filter, indicates 4° B., it is then concentrated to 20° B., and the now yellowish juice is allowed a fixed time for cooling and settling. It is next filtered cold, some of the separated flocculent albuminous bodies being soluble on warming, it is next filtered through animal charcoal, running off clear and colourless. The evaporation is now carried on to 40° B., in copper vessels. Solid maltose, containing 80 per cent. of sugar, is distinguished from glucose by not being hygroscopic, has a very agreeable sweet taste and an aromatic odour. The syrups mix without turbidity with water in all proportions, time showing no influence on their transparency.—F. L. T.

*On the Cultivation of Sugar-cane.* *Dingl. Polyt. Journ.* 253 [3] 136.

IN Pernambuco a fungoid disease of the sugar-cane has set in, whereby the stems are completely covered with the mycelium of a fungus; even the interior is filled up with it. According to De Bary it is *Schizophyllum commune* Fries.

W. Knop has made some analyses of ashes of diseased (I. and II.) and healthy (III.) canes, and the samples, dried at 100° C, contained:—

	I.	II.	III.
SiO <sub>2</sub> .....	810 .....	1065 .....	1150 .....
CaO .....	060 .....	245 .....	120 .....
MgO .....	162 .....	463 .....	225 .....
P <sub>2</sub> O <sub>5</sub> .....	070 .....	142 .....	120 .....
SO <sub>2</sub> .....	080 .....	107 .....	095 .....
Cl .....	289 .....	310 .....	313 .....
K <sub>2</sub> O .....	861 .....	915 .....	895 .....
Na <sub>2</sub> O .....	001 .....	015 .....	015 .....
Total ash ....	2'333 .....	3'262 .....	2'963 .....

Mn and Fe are not estimated, as the small amount cannot be separated in the presence of P<sub>2</sub>O<sub>5</sub>. The fluctuations of the constituents are not larger than usual, and no conclusion can be drawn from the analyses. The percentage of Cl is surprisingly high compared with other plants growing on land; the quantity of MgO is larger than that of CaO; the total ash of sugar-cane is smaller than that of any other cultivated plants. It therefore takes up from the soil less mineral matters than any known plant. Obviously the fungus has nothing whatever to do with the soil of Pernambuco, nor is it caused by useless or injurious admixtures to the manure.—S. H.

## XVI.—BREWING WINES, SPIRITS, Etc.

On Kephir. H. Struve. Ber. 17, 1364.

IN the Caucasus kephir has been used for some time as a drink and in medicine, and recently its consumption in Russia has made considerable progress. In Teflis also it is now the custom to take kephir medicinally, although its preparation in reference to the production and significance of the so-called kephir grains is still obscure. In the Caucasus kephir is prepared from milk, particularly sheep's and goat's milk, in peculiar leather pipes (burdinks) by adding to it kephir grains as ferment. The author has investigated the formation of this ferment and studied its properties. Since the augmentation of the kephir grains takes place when the milk is kept in leather pipes their growth must necessarily be affected by the milk constituents, and although it was *a priori* impossible to determine what changes the ingredients consumed during this process undergo, it was assumed that the substances belonging to the group of proteids would maintain their general type to a greater or less degree. By upholding this view the author was enabled to analyse the kephir grains, and investigate their properties more closely. 100 parts of the grains dried in the air gave—

Water .....	11'21
Fat .....	3'99
Peptones soluble in water .....	10'98
Proteids soluble in ammonia .....	10'32
Proteids soluble in potash .....	30'39
Insoluble residue .....	33'11

On digesting the insoluble residue in a dilute solution of potash, and subjecting it to microscopical examination it showed an intimate mixture of yeast fungi with the bacteria described by Kern under the name of *dispora caucasica*. The author concludes that only 33'11 p.c. of the total substance of the grains constitutes the active ferment. With kephir grains of the above composition a drink is prepared which, according to Podwisozy, contains only traces of alcohol after 24 hours' bottling, whilst in 48 hours it becomes brisk and contains a considerable amount of alcohol, the latter increasing still further after the lapse of another 24 hours. On subjecting the three drinks, characterised by the difference in the duration of the fermentation, to analysis it was found that although the percentage of casein was the same in each case, the latter was not completely soluble in a mixture of dilute ammonia and potash. In the solution of the casein of the kephir of one day's fermentation only traces of a precipitate were observed; the kephir two days old gave 0'05 p.c., and that three days old 0'22 p.c. of residue. Microscopical examination of the residues showed that yeast fungi only were present, bacteria or other forms of fungi being entirely absent. From this the author infers that the fermentation of the milk is dependent upon the growth of the yeast fungus (*saccharomyces mycoderma*,

*mycoderma urevriae* et vini) contained in the kephir grains, whilst the bacteria, *dispora caucasica*, take no active part in the fermentation. This conclusion is confirmed by the fact that the finished kephir is capable of fermenting fresh portions of milk. From the results of this investigation the author makes the following suppositions:—(1) During the fermentation the yeast fungus, under the influence of the power of vegetation and of osmotic laws, enters into animal as well as vegetable tissues, and disposes them to a variety of changes. (2) The development of the yeast fungus in the interior of organic tissues may, under favourable conditions, assume the character of a special process of augmentation. (3) The effects of such a process are increased when the evolution of carbonic anhydride is retarded. (4) Animal tissues into which the yeast fungus has entered exhibit in sugar solutions as well as in milk all appearances of vinous fermentation. They can therefore be used instead of kephir grains for the preparation of kephir. (5) The formation of the kephir ferment is the cause of a peculiar growth of the yeast fungus within the tissues of the leather pipes during the process of fermentation, its fungoid form being due to the development of carbonic anhydride. (6) The bacteria *dispora caucasica* must be regarded as residues of fibrils of the tissues of the leather pipes. These conclusions remain theses until they are confirmed by actual examination of the leather pipes used in the Caucasus.—D. B.

## XVIII.—SANITARY CHEMISTRY, DISINFECTANTS.

Action of Water on the Inner Coating of Tinned or Sulphurised Lead Pipes. Belohoubek. Chem. Centr. 15, 346.

AN examination of lead pipes tinned on the inside showed that the coating of tin varied from place to place from 1 to 0'1mm. in thickness, showing by means of an eye-glass or microscope longitudinal cracks, in some cases penetrating to the lead. This evil, already noticed from another point of view, has no little influence on the durability of the pipes, the water being simultaneously in contact with lead and tin, causing the lead more rapidly to oxidise and the oxide dissolving in the water. Tinned lead pipes with cracks have been shown to be more rapidly destroyed by water than lead pipes without any coating of tin. The examination of the tin coating, which was rubbed off so carefully as to leave a film of tin still on the lead, showed that it contained nearly 51 per cent. tin, the remainder being chiefly lead, the coating having a composition similar to common solder. Pipes coated internally with lead sulphide, according to Schwarz's method of treatment with liver of sulphur, showed that the coating was perfect, but scarcely 0'1mm. thick and in places blistered. Experiments with distilled water showed that sulphurised and tinned lead pipes seemed to resist its action in absence of air, whilst in the reverse case and on long use no small quantities of lead were found in the water (1 litre of distilled water took, in presence of air, in the open pipe in 24 hours at 18° to 21°, 1'839mgrm. lead from the sulphurised pipe, but only traces of lead and tin from the tinned pipe. Water and air kept at the same temperature 48 hours in the closed pipes, showed in the case of the sulphurised pipe 3'967 mgrm. lead per litre, with the tinned pipe 4'684 mgrms. per litre). When the water flowed through the pipes the result was more favourable, requiring the evaporation of 10 to 12 litres of the water to show the presence of tin and lead. Experiments with the very soft Moldavian water showed that the sulphurised lead pipe partly filled with water contained after 24 hours at 19° 7'274 mgrm. per litre; the water in the tinned pipe 5'144 mgrm. per litre. In the absence of air the sulphurised pipe showed 15'203 mgrm. lead per litre and the tinned pipe 7'831 mgrm. per litre.—F. L. T.

On the Continuous Absorption of Lead through Food Contaminated with it. A. Gautier. Chem. Centr. 15, 347.

IN preserved vegetables the lead present is derived chiefly from the solder used, which is rich in that metal,



the tinning of the tinplate never containing more than 1 per cent. of lead. In tinned vegetables obtained by Gautier in Paris, he found on an average 2.5 mg. of lead per kilo. The amount of lead increases on keeping. Gautier finding at the end of one year 1.2 mg. per kilo., after two years 2.1 mg. and after three years 4.2 mg., on an average. In tinned sardines he found 20 to 50 mg. of lead per kilo of the fish; the olive oil remaining in the tin containing a larger amount of lead. In a kilo. of *pâté de foies gras* there were 11.8 mg. of lead = 43 mg. of the oleate. Tinned lobsters contained as a mean 27 mg. lead. Gautier, contrary to Schützenberger and Boutmy, who found from 50 mg. to 1.48 g. lead per kilo of tinned beef for the navy, could find no trace of lead in American corned beef well tinned and soldered from the outside.

Drinking water takes up a very small quantity of lead on flowing through lead pipes, even when the pipes are treated with lime water; this quantity increases with the purity of and the amount of air dissolved in the water, and in the case of rain or distilled water may become dangerous. In artificial aerated waters, the author found in some cases 0.436 mg. lead per litre, the lead apparently arising from the solder of the syphon. Water, wine, vinegar, beer, after standing for a long time in vessels of crystal glass contained very little lead. Tin vessels were found more dangerous, those used in the civil hospitals in Paris are allowed to contain 10 per cent. of lead, whereas those in the military hospitals may not contain more than 5 per cent.

Gautier concludes from his experiments and those of his predecessors that even if the continued use of water and beverages which have passed through lead pipes, and of tinned vegetables, produces no dangerous effects, yet this is not the case with tinned meat or fatty foods, which may contain considerable quantities of the poisonous metal. Leroy de Mericourt on the other hand points out that lead poisoning very seldom occurs in the navy, where, however, tinned food, and especially sardines, are frequently consumed. The author insists that the greatest care should be taken in the preparation and preservation of foods to prevent the possibility of the introduction of lead. —F. L. T.

*On the Prevention of the Pollution of Streams by the Waste from Industrial Processes.* Dingl. Polyt. Journ. 253, 84, 1884.

In the *Vierteljahrsschrift für Gerichtliche Medicin* 1883, 121 and 298, Wolff records some observations he has made of processes employed in England and Scotland for preventing the pollution of streams by factory waste waters. A Scotch paraffin oil works, which previously allowed their waste to flow into the Midealder, with the result that the water of that river contained 54 mg. of paraffin oil per litre, now adopt the plan of evaporating their waste liquors after they have been allowed to deposit matter suspended therein. The vertical retorts employed are provided at their lower end with a conical tubulure, which dips for 15 or 20 cm. under the water to be evaporated; the latter passes partly into the retorts and partly into the atmosphere. Waste liquors from paper mills are in several establishments efficiently purified by lime; processes of filtration, and of precipitation by means of ferric chloride and lime are also being adopted. As recorded in the *Civilingenieur*, 1883, page 229, the *Technische Deputation des K. Sachsischen Ministeriums des Innern* has been led to report that whilst the English River Pollution Prevention Act of 1876 could hardly be put in force *in toto* in Saxony, it contains two clauses which would, if made the subject of legal enactment, effect public benefit. According to these clauses, the introduction into a river or stream of solid waste from industrial processes, and of poisons and noxious liquors, is constituted an offence; if, however, it can be shown that the manufacturer employed the most efficient processes known for the purification of his waste, the offence is no longer chargeable. Since solid matters in suspension are removable by filtration, the regulations as to their introduction into rivers and streams should be strictly enforced; on the other hand, the clause which permits the manufacturer to purify his waste liquors by the most efficient processes known

will permit of some indulgence being shown during the first periods of working the law, and may be expected to be conducive to the improvement of old and invention of new processes. To ensure a uniform administration of the law throughout Saxony, it is recommended to appoint a Commission of Chemists and Technologists, whose duty it shall be to advise the local authorities on technical questions, to undertake investigations, and to confer with manufacturers as to methods of purification. It is suggested in conclusion that where one stream is polluted by several establishments in close proximity to each other, that these firms should be urged, or even coerced, to combine in providing some general means of preventing that pollution. By such combination the means adopted would be more efficient and less expensive than in the case of individual action. —W. D. B.

## XIX.—PAPER, PASTEBOARD, Etc.

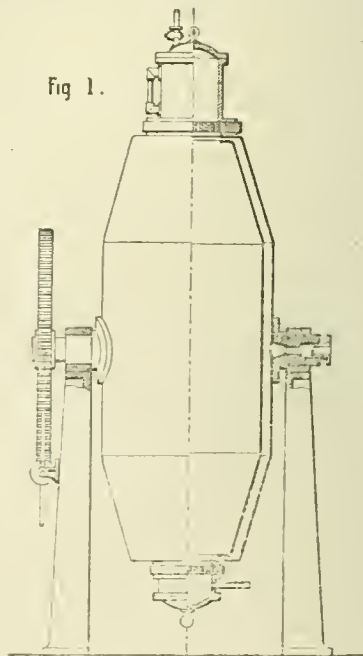
*Chemical Wood Pulp-Sulphite Processes.* Ding. Polyt. Journ., 252, 323.

(a) J. A. Graham (Eng. Pat. 5365, 5366, 5367, 5368—1882) has suggested a number of modifications of the now well-known processes of preparing wood pulp by means of the sulphites. In the first of the patents cited above, he directs the introduction, in the first instance, into the digestors of solutions of the neutral sulphites, and after expelling air and carbonic acid from the apparatus, of sulphurous acid under pressure, in quantity somewhat less than sufficient to convert them into bisulphites. In patent 5366 he directs the addition of a nitrate, in the operation of bleaching the pulp with bleaching powder, with the object of preventing the formation of hydrochloric acid, and of allowing a high temperature of the mixture without injury to the pulp.

(b) Eng. Pat. 5367 (also Ger. Pat. 23718, 1882), describes a special method of covering iron plates for the manufacture of the digestors with lead, which has been already reproduced (*Ibid.* 251-70). The process consists in pouring the molten lead upon the iron plates previously treated with zinc chloride, and offers therefore no novelty. In working up these plates into the digestors, they are first bolted together in the ordinary way, the junction of the edges being then completed by means of lead. The method is simple, and appears to be effective and economical (cf. *Papierzeitung*, 1883, 434).

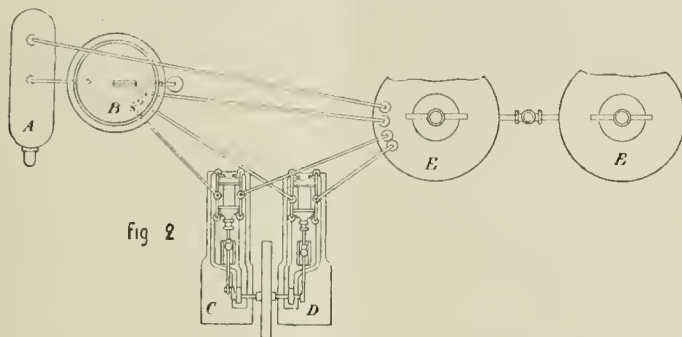
(c) Patent 5365 describes the digester of special construction, figured below. This consists of a cylindrical

Fig 1.



body, with dome-shaped moveable extremities. The contents of the digester are heated by means of a jacket of steam.

(d) K. P. Pictet and G. L. Brelaz (Ger. Pat. 26, 331, 1883) condemn the various sulphite processes hitherto adopted on account of the high temperature ( $120^{\circ}$ – $140^{\circ}$ ) at which the digestion requires to be conducted, the effect of which is to convert the resinous constituents (of wood) into tarry products, the presence of which in the pulp renders the subsequent bleaching process the more difficult. By employing an aqueous solution of sulphurous anhydride, the disintegration of wood may be effected at low temperatures, the most convenient conditions having been found to be a strength of the acid solution represented by 100–150 gr.  $\text{SO}_2$  in ll. water at the temperature of  $80^{\circ}$ – $90^{\circ}$ , at which the pressure (of  $\text{SO}_2$ ) is between 5 and 6 atm. The apparatus is represented in figs. 2 and 3. E is the digester provided with the worm S, by means of which and a current of steam the contents of the digester are heated, and a manhole at I.



The  $\text{SO}_2$  is introduced from the reservoir A by means of a system of tubes and cocks; or it may be injected as it is evolved, and without previous condensation, by means of the pump C. It is advantageous to render the digester containing its charge of wood vacuous before introducing the acid solution. This is effected by means of the air pump D. The wood is thus thoroughly impregnated with the solution, and its action is thus evenly and rapidly exerted throughout the mass of the wood. The liquid at the close of the operation contains the non-

(f) D. K. Francke, whose process has been already described (*Ibid.* 1883, 24924) has introduced this method into America (U.S. patent 295868, March 28, 1884) with certain modifications. The digestors described in this specification are simple rotary cylinders 12.5m. long by 2.2m. diameter, the inner surface being provided with projections, with the object of preventing the balling of the pulp. For 1,000k. wood, 900–1250l. of sulphite solution are required, *i.e.*, of a solution of acid calcium sulphite of 4.5 B°. The temperature recommended is  $150^{\circ}$ .

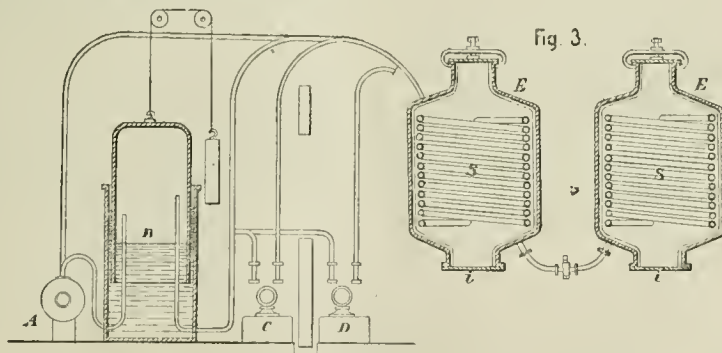
(g) C. Kellner and H. Ritter (French patent 157754, 1883) describe a special process for the preparation of sulphite solution, and a digester for the preparation of wood pulp. The reproduction is unaccompanied by figures, and the original specification should therefore be consulted.

(h) R. Blitz (French patent 155014, 1883) disintegrates wood by means of a solution of sodium sulphide and hydrate, together with ammonium vanadate in small quantity.—C. F. C.

*Paper from Pulp prepared by the Sulphite Process.*—*Papierzeitung*, 1884, p. 938.

PACKING paper made from pulp, which has undergone a treatment with sulphite, is likely in time to cause injury to materials packed therein, by reason of the sulphurous acid compounds it contains. It is recommended to treat such paper with a solution of bleaching powder to destroy the residual sulphurous acid.

W. D. B.



cellulose of the wood in the form of soluble derivatives, together with the excess of sulphurous anhydride, which is easily recovered. It has been found that the same solution may be used for several successive charges of wood, and the digestors are therefore placed in serial connection, as represented in the figure (EE).

(c) The Oesterreichische Verein für Chemische u. Metallurgische Production have patented a process (Germ. pat. 25485, 1882), which consists in digesting wood with a solution of sodium sulphide of 10° B. (30 pts.  $\text{Na}_2\text{S}$  to 100 pts. wood) at a pressure of 5–10 atm. for 10–6 hrs. A subsequent treatment of the disintegrated wood by the ordinary processes yields a pulp of good quality.

*Improvements in or connected with the Bleaching of Paper Pulp, or other Fibrous or Textile Materials or Fabrics.* Eng. Pat. No. 5160. Oct. 30, 1883. John Henry Johnson.

THE process patented is chiefly applicable to bleaching paper pulp. It consists of two stages. In the first, chloride of sodium or potassium is decomposed by an electric current in presence of lead in such a way as to obtain a solution of the caustic alkali and chloride of lead. The second stage consists of decomposing the chloride of lead in such a way as to obtain chlorine. The operation is performed in an ordinary rag engine, the bottom of which is almost entirely covered with a plate of lead, constituting the negative electrode. Over

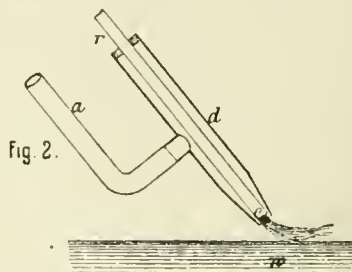


this is arranged an insulated sheet of wire gauze. The positive pole is placed above the gauze, insulated from it, and consists of a plate of carbon, platinum, or other material not acted upon by chlorine. The lead chloride from the first operation, mixed with water and a little hydrochloric acid, is placed in the engine. The electric current causes nascent chlorine to be generated at the positive pole; this dissolves in the liquid, and bleaches the materials under treatment.—E. J. B.

*Improvements in the Manufacture of Paper Pulp and in Apparatus therefor.* Eng. Pat. No. 6376. April 15, 1884. Henry Harris Lake: a communication from Goldsberg Horden Pond and Edmund Alonzo Morse.

THIS invention relates to the manufacture of mechanical wood pulp. The apparatus consists of a strong cylinder fitted with a steam-tight cover. It is provided with a central shaft, bearing arms on which are supported rollers which, when the shaft revolves, press against the inner surface of the cylinder. The sawdust or other material mixed with water, from which it is desired to make pulp, is charged through a hole in the cover of the cylinder, which is then closed, and steam at a pressure of about 50lbs. per square inch is let in, and the shaft carrying the rollers made to revolve rapidly. The disintegrating action is assisted by the addition of a small percentage of alkali or alkaline earth. Descriptive drawings accompany the specification.—E. J. B.

burning over water. The air is forced into a tube *r* (Fig. 2), which is drawn out to a small orifice at *e* while gas enters *a*. It is ignited at *c*, and the flame blown



deeply into the mass of water *w*, care being taken that the pipe *d* inclines to the surface of the water at an angle of 30° to 45°.—S. H.

## XXII.—GENERAL ANALYTICAL CHEMISTRY.

*A Drop Method of Qualitative Analysis.* H. Hager. Chem. Zeit. 1884, page 823.

THE ordinary methods of testing medicinal preparations, which as a rule require a large amount of substance, may to a considerable extent be replaced by a method in which only single drops of solution are worked with. The following reagents are prepared for this so-called drop method: *Litmus paper*, blue and red; *turmeric paper*; *indigo carmine paper*, which becomes yellow with warm nitric acid and caustic alkalis, but not with ammonia; *rosaniline paper* for alcohol testing; *potassium ferrocyanide paper* as a reagent for ferric salts (blue), copper (dark brown), uranium (dark brown), gold (greenish brown), platinum (brownish green to red), thallium, vanadic acid (yellow); *potassium thiocyanate paper*, coloured strongly yellow by acid solutions of bismuth nitrate, bluish-black by copper salts, red by gold solution, white by mercuric nitrate, black by mercurous nitrate, and blood-red by ferric salts; *potassium iodide paper*, coloured red by mercuric, green by mercurous salts, yellow by lead solutions; in presence of chlorates, a solution acidified with dilute sulphuric acid becomes yellow if a strip of this paper be immersed therein and the whole gently warmed; *mercuric nitrate paper* when moistened is a delicate reagent for ammonia gas, which blackens it, caustic alkalis or their monocarbonates colour it greenish-brown to black, whilst the bicarbonates do not alter it; *silver bichromate paper* is coloured yellow by free hydrochloric acid. The author mentions other reagent papers employed less frequently than the above. A drop of solution is placed upon these papers, and the colour changes carefully observed. Arsenic is tested for by the aid of a strip of sheet brass.

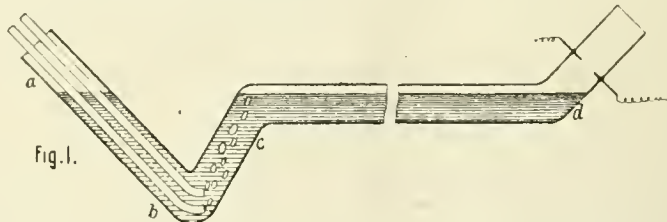
## XX—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Preparation of Hydrogen.* Dingl. Polyt. Journ. 253, 92, 1884.

BERARD recommends Egasse's process for the preparation of hydrogen from zinc and hydrochloric acid. The hydrogen is chiefly employed for filling balloons, and costs 0.8 mark per 1 cubic meter. The residual chloride of zinc may be employed for purposes of disinfection, wood preservation, and the like.—W. D. B.

*Preparation of Hydrogen Peroxide.* M. Traube, Breslau. Germ. Pat. 27163, 1883.

THE author has observed that a CO flame produces no trace of  $H_2O_2$  in a dry bottle containing air, but great quantities of that compound, if it be conducted over water, or burnt in close contact with it. Even the H flame, if it be brought in close contact with  $H_2O_2$ , produces much  $H_2O_2$ . This compound pre-exists in the flame, and is destroyed by the heat unless it is cooled by water. To prepare peroxide of hydrogen, gas mixed



with air is allowed to enter a long horizontal pipe filled so far with water as to leave only a narrow channel for the gases. (The so-called "water-gas" is the best form of gas to employ as above.) Fig. 1 shows a tube filled with water, and bent at an angle at *b*. In the limb *a* are the two tubes for gas and air. These are made to bubble through the water, pass over it, and are exploded at *d*. According to another suggestion, water in fine sprays is blown through a flame, and thus charged with  $H_2O_2$ . Or a stream of air is pressed through a flame

A hydrochloric acid solution of arsenic is reduced by the addition of oxalic acid and a drop of this solution placed upon the brass; the plate is rapidly dried, washed with water, and any stain caused by the drop examined; in presence of arsenic the stain is of a dark permanganate colour. This reaction is very delicate. When neither reagent papers nor the brass plate can be employed, droplets each of the solution and reagent are placed upon a small disc of glass, mixed, and any changes observed.—W. D. B.

*Detection and Estimation of Ammonia in Animal Fluids.*

By J. Latzchenberger. Monatsh. f. Chem. 1884. p. 129.

VARIOUS methods have been proposed for the determination of ammonia when accompanied by proteid bodies, notably those of Heintz, Schlösing, E. Salkowski, and Bosshard. These are fully discussed by the author, and pronounced more or less untrustworthy. The new method which he has devised is very fully described in this paper, as are also the various stages through which it was perfected. It is grounded upon the observation that ammonia was completely precipitated by Nessler's reagent from a solution in which urea was also present. From this precipitate, after washing with the diluted Nessler reagent by which it was entirely freed from urea, the ammonia was isolated by distillation with barium hydrate, after first removing the mercury by treatment with hydrogen sulphide in presence of hydrochloric acid. Although, however, good results were obtained with a solution containing urea and ammoniacal salts only, *i.e.*, artificially prepared, it was found that with such natural mixtures as urine and water the method no longer gave accurate results. However, by means of Wanklyn's colorimetric method, preceded by preliminary treatment of the liquid with copper hydrate, it was found that the ammonia could be accurately determined in the filtrate from the latter. The general outline of the method is as follows:—Into a beaker of about 120 cc. capacity, 20 cc. of a cold saturated solution of copper sulphate are run, and the whole is weighed. 20 cc. of the solution to be tested are then added, and a second weighing taken, which gives the weight of the latter. The solution is then very carefully neutralised with baryta water, and introduced into a litre flask, the volume being made up with the rinsings of the beaker, afterwards with pure water. It is then filtered and tested with the Nessler solution comparatively with a standard solution of ammonia chloride in the usual way. The author gives full details of the method, together with the precautions necessary to be observed in special cases; also results of experiments with blood, urine, milk, and bile, which leave little to be desired in point of accuracy. In applying this method to the study of human urine, the author has found that it contains appreciable quantities of ammonia. Thus in one instance, which he cites, of a normal urine of sp. gr. 1.021, voided in the afternoon, he found that  $\frac{1}{4}$  of the total nitrogen was present in the form of ammonia. The following are the absolute quantities of ammonia estimated by the author in normal specimens of various natural fluids:—

	Per cent.
Human urine, sp. gr. 1.021 .....	0.05550
Dog's urine, sp. gr. 1.018 .....	0.08038
Cow's milk .....	0.02106
Ox's blood .....	0.00781
Ox's bile .....	0.00283

Many other determinations have been made in specimens of these same fluids, of which the results have not sensibly varied from the above. The author calls attention to the physiological bearings of the determinations of ammonia in milk, blood, and bile, which are from animals of the same species. In conclusion, the author points out that previous determinations of ammonia by less trustworthy methods still retain their value in so far as the numbers are relative, when the error attaching to the method is constant.—C. F. C.

## Monthly Patent List.

### ENGLISH APPLICATIONS. 1881.

11513 T. Nordenfelt, Westminster. Improvements in apparatus to be employed in burning naphtha or other volatile liquid, applicable especially for melting metals which are difficult of fusion, but also useful for other purposes. Communicated by L. Nobel, St. Petersburg, C. G. Wittenstrom, Motala, E. Faustman and P. Ostberg, Stockholm. August 22

11517 W. D. Curzon and G. Jones, London. A substitute for fullers' earth. August 22

11518 W. D. Curzon and G. Jones, London. A new or improved manufacture of a compound suitable for scouring and for other purposes. August 22

11537 A. M. Clark, London. Improved apparatus for use in treating wood and other ligneous matters by means of hydro-

chloric acid gas, in order to obtain glucose therefrom. Communicated by L. J. A. Lespermont, Paris. Complete specification. August 22

11586 W. A. Barlow, London. New or improved process for the manufacture of extracts of tannin, and apparatus therefor. Communicated by La Société Civile d'études sur les Procédés Nouveaux de Fabrication Perfectionnée d'Extraits Tanniques, Paris. August 23

11615 J. C. W. Stanley, London. Treatment or preparation of animal charcoal for the manufacture of blacking and for other purposes, and apparatus therefor. August 25

11622 D. C. MacVail, Glasgow. Rendering aseptic the air entering vessels holding milk or other fluids. August 26

11628 B. Hammond, Guildford. Coppered blue for prevention of contagious and other diseases. August 26

11639 J. Hopkinson, London. Improvements in lubricants. August 26

11611 E. J. Leveson and J. W. Slater, London. Improvements in the preparation of agents to be used in the treatment of sewage and other liquid or semi-liquid putrescent or putrescible matters, and improvements in the treatment of such matters. August 26

11651 D. O. Francke, London. Improvements in the method of and in boilers or apparatus for treating wood pulp and other vegetable fibre. Complete specification. August 26

11655 A. Mitscherlich, London. Improvements in boiling with sulphites. August 26

11660 H. H. Lake, London. Improvements in and relating to the coating or covering of wood or other surfaces, and paint or composition therefor. Communicated by L. Brown, United States. August 26

11661 H. H. Lake, London. An improved method of and apparatus for purifying water for use in steam boilers. Communicated by G. H. Knott, United States. Complete specification. August 26

11701 H. M. Whitehead, London. Improved means or mode of extracting gold and silver from their ores, and in apparatus connected therewith. August 27

11705 J. W. Woore, London. Improvements in the manufacture of alloys of copper and zinc. August 27

11711 A. Feldmann, London. Improvements in the process of manufacturing ammonia. August 27

11735 H. Gardner, London. A new or improved liquid compound for extinguishing fire and subduing flame. Communicated by R. A. Saffield, United States. August 28

11718 T. Bayley, Birmingham. An improved method of treating human bodies for the prevention of putrefactive change. August 29

11775 H. Gaskell, junior, London. Improvements in the purification of bicarbonate of soda, and apparatus therefor. August 29

11780 T. Hawksley, Brighton. The more practical and practicable disposal of human excretions and other refuse organic matters with a view to their agricultural value, the public health, and the abolition of the use of water in the removal and conveyance of such matters. August 30

11805 P. M. Justice, London. Improvements in the manufacture of phosphates. Communicated by S. G. Thomas, Paris. August 30

11808 R. W. S. Griffith, London. Improvements in the manufacture of gunpowder. August 30

11809 C. J. Ball, London. Improved means of and apparatus for effecting the separation of diamonds, gold, tin, and other minerals from earthy and other matters. August 30

11816 A. Mitscherlich, London. Improvements in the manufacture of paper pulp. Aug. 30

11827 J. Simpson and E. W. Parnell, Liverpool. Improvements in the treatment of ores containing sulphide of antimony for the purpose of obtaining the said sulphide in a concentrated form. Complete specification. September 1

11828 E. W. Parnell and J. Simpson, Liverpool. Improvements in the treatment of ores containing sulphide of antimony for the purpose of obtaining the said sulphide in a concentrated form. Complete specification. September 1

11810 W. W. Daw, Chadwell Heath, Essex. Solidifying urine. September 1

11838 W. R. Lake, London. Improved incombustible compounds to be used for the protection of wood or other materials. Communicated by La Société Vve. Wendt et Hérard, Nancy, France. September 1

11866 J. Meikle, junior, Glasgow. Improvements in purifying sulphuric and sulphurous acids and their salts. September 2

11830 F. G. Riley, London. Improvements in apparatus for carbonating liquids or semi-fluids. September 2

11901 W. Astrop, London. An improved process of deodorizing, disinfecting, precipitating, drying, and pulverising the solid portions of sewage to render it a marketable manure. September 2

11930 J. Connor, Glasgow. An improved black pigment. September 3

11942 P. Spence, New York. An improved blue colouring compound for use in the laundry and for dyeing and other purposes. September 3

11947 H. L. Pattinson, junior, London. Improvements in the manufacture of hydrates of barium and strontium from their ores. September 3

11955 J. C. W. Stanley, London. Improvements in the treatment of starch cake and apparatus therefor. September 3

11967 R. de Soldenhoff, London. Improvements in apparatus employed in the treatment of coal in order to obtain coke and bye-products therefrom. September 3

11971 G. Jones and J. C. Bromfield, London. Improvements in the treatment of sewage and manufacture of manure therefrom. September 3



11982 J. Hamiltan, Derby. Improvements in sugar machinery and crystallization of fluids. September 4  
 11987 G. T. Beilby, Glasgow. Improvements in separating solid paraffin from oils. September 4  
 12008 E. Edwards, London. Improvements in artificial stone and in the method of making blocks, statues, and other objects of such artificial stone. Communicated by T. Grundmann, Hirschberg. September 4  
 12041 H. Herrenschmidt, London. Improvements in the extraction of cobalt, nickel, and manganese from their ores, and of nickel from its ores, when such ores are found in a similar condition to these of New Caledonia. Complete specification. September 5  
 12051 W. D. Curzon and G. Jones, London. Improvements in the manufacture of alum. September 5  
 12097 J. Maclaren,昌平, Fifeshire. A composite paving block. September 6  
 12105 J. C. Mewburn, London. Improvements in the manufacture of champagne or sparkling wines. Communicated by La Société Anonyme "La Vinicole," Paris. September 6  
 12159 J. Clare, Penketh, Lancashire. Improvements in packages for containing caustic soda and other articles, and in the lids or covers for the same. September 9  
 12171 W. Arrol and J. Meikle, Glasgow. Improvements in apparatus for making salts of ammonia. Complete specification. September 9  
 12182 J. E. Atwood, London. Improvements in process of purifying molten iron or steel. Complete specification. Sept. 9  
 12186 J. F. Gent, London. Improvements in the art of manufacturing cerealine, a product obtained from Indian corn. Complete specification. September 9  
 12193 C. W. Knowles, London. An improved process of preparing sulphate of lime for use in paper making. Sept. 9  
 12246 F. Wilkins, London. Improvements in asphalt pavement. September 10  
 12248 D. Hepworth, London. Improvements in dyeing. September 10  
 12262 S. P. Wilding, London. Improvements in the manufacture of iron, steel, and other metal. Communicated by H. W. Kasten and S. Hein, Hannover. September 10  
 12280 A. Parkes, London. Improvements in the production of compounds capable of being employed for the coating or covering of metallic, animal, vegetable, or other surfaces. September 11  
 12286 P. Dew, Glasgow. Improvements in retorts for distilling shale, coal, and other substances. September 11  
 12292 E. Luck and F. B. W. Roberts, London. Improvements in the manufacture of explosives and in the treatment of materials to be employed in the said manufacture. September 11  
 12307 F. M. Lyte, London. A process for the manufacture of sugar from sea-weed and alcohol therefrom, and the utilisation of the residues. September 11  
 12317 G. I. J. Wells, Liverpool. Improvements in the treatment of complex ores or solutions. September 12  
 12326 W. T. H. Carman, London. Improvements in glass starch. Communicated by W. Zwick, Neumühle-Albersweiler. Complete specification. September 12  
 12340 H. Simon, London. An improvement in coke ovens. September 12  
 12345 S. Deedman Cox, London. An improved method of and apparatus for purifying or disinfecting sewage and collecting the ammonia and other substances contained therein to serve as manure. Complete specification. September 12  
 12346 W. R. Lake, London. Improvements in the manufacture of paper pulp, and in apparatus therefor. Communicated by C. S. Wheelwright and G. E. Marshall, United States. Complete specification. September 12  
 12372 W. H. Beck, London. An improved process for colouring paper in endless rolls, and in apparatus to be employed therewith. Communicated by L. Piette, Pilsen. Complete specification. September 13  
 12379 C. Kingsford, London. Improvements in and relating to apparatus chiefly designed for utilising the waste heat from coke ovens or other furnaces for evaporating, calcining, and other purposes. September 13  
 12380 E. Nienstadt, Berlin. Improvements in printing inextinguishable metallic deposits upon ivory, bone, horn, nacre, and other organic substances. September 13  
 12381 A. Fullerton, London. Improvements in gas-producer furnaces. September 13  
 12385 W. Clark, London. An improved process and apparatus for bleaching waste-paper, whether printed or written on. Communicated by E. A. D. Guichard, Paris. September 13  
 12398 C. Lytle and J. J. Eastwick, London. Improvements in the treatment of saccharine solutions for the purpose of clarifying the same. September 15  
 12403 C. Lytle and J. J. Eastwick, London. An improved process for purifying water contaminated with impurities, such as sewage waters, river waters, or the like. Sept. 15  
 12410 T. W. Bassett, Mumford, and R. Moodie, London. Improvements in apparatus for the concentration of sulphuric acid, or for analogous purposes. September 15  
 12421 W. L. Wise, London. Improvements in obtaining hydrochloric acid gas from a solution of hydrochloric acid. Communicated by Messrs. Solvay and Co., Brussels. Sept. 15  
 12425 H. Kettmann, London. An improved process for the manufacture of asphalt. September 15  
 12427 T. Routledge, London. Improvements in boilers or digesters to be employed in the treatment of paper-making materials or for analogous operations. September 15  
 12428 J. Yate Johnson, London. Improvements in treating ramie or china grass or other like fibre-yielding plants for the purpose of separating the fibrous portion from the boon and rind, and in the apparatus employed therein. Communicated by Joseph A. Kanick, Paris. September 15

12435 W. Clark, London. Improvements in tanning skins and in the manufacture of compounds therefor. Communicated by Prof. E. Hermann, Vienna. September 15  
 12456 W. H. Beck, London. Improvements in apparatus for manufacturing hydrocarbon gas. Communicated by W. F. C. Mason McCarty, St. Petersburg. Complete specification. September 16  
 12457 L. A. Groth, London. Improvements in the treatment of petroleum. Communicated by T. N. Fiesch, Annweiler. September 16  
 12469 A. Fesca, London. Improvements in the manufacture of sugar, and in apparatus therefor. September 16  
 12471 J. P. Roe, London. Improved apparatus for the distribution of gas and air in regenerative steel melting, glass melting, and other furnaces wherein gaseous fuel is used for heating the same. September 16  
 12472 C. H. Preibisch, London. Improvements in dyeing fabrics aniline-black, and in apparatus therefor. September 16  
 12481 A. M. Clark, London. Improved apparatus for the manufacture of gas. Communicated by A. and T. Henning, United States. Complete specification. September 16  
 12483 O. Brawn, London. An improved method or process for separating salts from solutions containing the same. September 16  
 12486 T. Freeman, junior, and C. Eardley, Hanley, Staffordshire. Machinery and method of manufacturing hollow bricks and other hollow clay or plastic building materials. Sept. 17  
 12490 H. Grandage and R. Elliot Steel, Bradford, Yorkshire. An improved process in dyeing with substances containing tannin or tannic acid or gallotannic acid. September 17  
 12498 J. C. Mewburn, London. Improvements in processes and apparatus for dividing and distilling crude petroleum, and for refining the products obtained. Communicated by The Halvorsen Process Company, United States. Complete specification. September 17  
 12543 S. Puplett, Knowle, Warwickshire. Improvements in apparatus for compressing the condensable gases. Sept. 18  
 12556 J. Heaton, London. Improvements in converters employed in the manufacture of iron and steel. September 18  
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# THE JOURNAL OF THE Society of Chemical Industry:

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## NOTICES.

In reply to numerous inquiries the Secretary begs to inform Members that the first six numbers of the Society Journal for 1882 have now been reprinted, and can be obtained on application, while the orders already received are being executed with every dispatch. For the information of those desiring to have their copies bound, it may be convenient to know that the index and title page of Vol. I. are to be found in the July number, 1883.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 43, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

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#### ERRATUM.

Dr. Chas. A. Burghardt, Lecturer on Mineralogy at Owens College, requests us to state that he has not changed his address, as erroneously stated in the September number. Both addresses are correct.

### London Section.

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MEETINGS, SESSION 1884-5—First Monday in each month (unless otherwise indicated).

On November 3rd, Mr. W. H. Deering, F.I.C., F.C.S., will read a paper on "Some Points in the Examination of Tallow and some Commercial Oils, especially for Acidity." And Mr. W. S. Squire, Ph.D., will make some supplementary remarks to his Paper "On the Processes concerned in the Conversion of Starch into Alcohol and their Relation to Brewing and Distilling."

The Discussion on the Papers, and those of the Section published in the July and subsequent numbers of the Journal will be taken.

December 1st, Mr. J. M. Thomson, F.I.C., F.C.S., will read a Paper "On Photography, for those engaged in Industrial pursuits."

### NOTE ON BAKU PETROLEUM.

BY W. H. DEERING, F.I.C., F.C.S.

THE following note of an examination, made some months since, of a special distillate from Baku petroleum may be of interest to chemists connected with the mineral oil industry. It was desired to have a distillate boiling between 200° and 300°C.—a "light-house oil" in fact, but the material contained also a portion distilling below 200°C. It was almost colourless, and showed blue fluorescence. Its specific gravity at 15°5C. was 0·8225 (water at 15°5C. being 1). On distilling it, nothing came over until 166°C. (the temperature of the vapour being taken, and the barometer showing 767mm. at 0°C.), and the following percentages of distillate were obtained:—

°C.	Percentage by Weight.	Sp. gr. (at 15°5C).
166°—200°	31·0	0·8078
200°—250°	49·9	0·8243
250°—300°	13·1	0·8460
Above 300°	3·0	—
	100·0	

No sulphurous oxide evolved during distillation. The flashing-point of the original material, with the Abel-apparatus, is 50°5C. (123 F.), barometer being

as above; and it continued to burn in an open vessel at 63°C. (145°F.). The 200°-250°C. fraction flashed at 60°-5C. (141°F.), which shows the lowering influence of the 166°-200° fraction.

The higher specific gravity of the constituents of Baku petroleum, compared with that of those of the same boiling point from American petroleum, has often been pointed out; but the fractions of this Baku petroleum are also of higher specific gravity than similar fractions of the Scotch paraffin light-house oils that I have examined.

This petroleum was devoid of rotatory power to plane-polarised light; using a length of 200mm., and Hofmann's form of the Laurent polarimeter, I saw no rotation with the original material, nor with either the first or the second fraction. (A rotation of  $\pm 0.30'$  would have been just visible.)

Markownikow and Ogloblin, in their account of their examination of Caucasian petroleum, from the peninsula of Apseheron, also mentioned the absence of rotatory power in the petroleum, using a length of 200mm.

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Notices of papers and communications for the meetings to be sent to Edward Geo. Ballard, Queen's Park, St. Helens.

The next meeting will be held at the University College, Ashton Street, on Wednesday evening, November 5, 1884, at Seven o'clock.

The following communications have been promised: (1) Mr. John Hargreaves—Visit to the Rio Tinto Mines; (2) Mr. A. Norman Tate—Instruction and Examination in Chemical Technology; (3) Dr. Hurter—The advisability of adopting the Metric System of Weights and Measures in this country.

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MEETINGS, SESSION 1884-5, Oct. 9th, Nov. 6th, Dec. 4th, 1884; Jan. 8th, Feb. 5th, March 5th, 1885.

*Meeting, October 9th, in the Lecture Theatre of the College of Science.*

## INAUGURAL ADDRESS OF THE CHAIRMAN, MR. J. C. STEVENSON, M.P.

IN opening another session of the meetings of the Newcastle Section of the Society of Chemical Industry, I feel with the committee that the great success of the annual meeting of the whole Society, which was recently held on the Tync, will create expectations in the other Sections of a more than usually interesting programme of papers and discussions during the season now beginning, and I hope we shall not be disappointed. Among so many members occupied in conducting the practical operations of chemical industry, there must be not a few who have given attention to, and acquired experience in, many various problems which they have either solved or failed in solving; and the record of such failures would furnish interesting information and subjects of discussion at our meetings. Indeed, I am inclined to think that the failure of well-intentioned attempts at improvements or economies should be as carefully recorded as the successes, or even more so, because if the failures are hopeless, others will be prevented from repeating them, and if they are due to want of perseverance or of skill, it may be the happier lot of subsequent experimenters to bring them to a successful issue. Moreover, there is no monopoly value in a failure, and no reason why it should be kept secret from the world. Nor is there any discredit in having made the attempt, for, to vary a hackneyed line, it is better to have tried and failed, than never to have tried at all. There is a laudable spirit of improvement, which I should seek by all means to encourage, and which has been of much use in late years in reducing the cost of production in some of our hard-pressed industries; so that the cost of production has been reduced to an extent which some years ago would have appeared incredible. When the severity of the competition of novel modes of manufacture, such as the alkali trade is now undergoing, compels the introduction of some kind of economy, the first impulse often is to try whether this cannot be effected by the substitution of hand labour by machinery. My view is that this tendency is to be resisted—not obstinately against reason and experience—but yielded to only after ample demonstration of the clear preponderance of advantage, considering first-cost and current expenditure of the mechanical appliances. Fortunately we are not all so cautious and conservative as I have indicated, and we are under great obligation to those manufacturers who have been enterprising enough to lay out large sums of money in attempts in this direction, which it is the more courageous in them to do when industry is in a transition state, and when one cannot tell what modifications may be required to adapt it to the only too rapidly altering circumstances of these progressive days.

Not caring whether or not the rest of this paper may be treated as an excuse for the more cautious policy which many of us have pursued, I propose to discuss the question of the limits of the profitable application of machinery in place of hand labour in some branches of chemical industry. The most striking instance of a successful use of machinery is the revolving-ball furnace; but this was more than a new mechanical mode of working, for it involved



an alteration of the furnace itself. Balling is really a crucible operation, and the greater depth of the mass of material in the cylinder enables it to be more effectually treated than when spread in thin layers on the floor of the old hand-furnace. The barrel shape of the large furnaces recently erected still further approximates to the crucible principle. The revolving cylindrical furnace has the further great advantage of the brick lining alternately receiving heat from the flue and giving it up to the materials as it revolves. There is no cooling from an open working door, and no burning of the materials as took place when the workman was resting between his "spells" of turning over the charge. The steam power required is comparatively small, and there are no disadvantages worth reckoning on the other side, and the early difficulties connected with the wearing of the wheels and rings have been overcome by the use of steel and other improvements. But with all this, I should question the economy of such machinery in a manufactory in which two or three hand-furnaces would be sufficient for the work. Indeed, I know of a very well managed and successful little caustic soda work in Lancashire which uses bought salt-cake, in which there is not a single steam engine, except for feeding the boilers, and in which steam jets are used for raising liquids instead of pumps.

I have always considered the old hand-carbonating furnace much more scientific than the mechanical carbonator which Mr. McTear introduced. The former was a reverberatory furnace in the strictest sense, where the "black salts" were heated from the body of flame which passed slowly over them without touching them, while the almost constantly open door allowed a sufficient supply of air to pass into the furnace and come in contact with the salts, by which, and by the carbonic acid in the furnace, the salts were carbonated and oxidised. The labour required was continuous, but not severe, and the consumption of fuel was less than in the mechanical furnace, without reckoning that required for the steam power for the latter, while the repairs were very slight. The McTear furnace makes no provision for such a supply of air as I have described, and the salts are heated by a rapid flame in near contact with them, so that a complete oxidation of the salts is very difficult. The advantages are, however, that they are finished at a higher heat, and the density is increased while the colour is improved. There is also a saving of hand labour. The great heat, with the constant exposure to it of the central plugs and stirrers, necessitates considerable repair, and the cost of lubricating hot machinery is necessarily considerable. While there is a preponderance of advantage over the old method, it is much less than in the case of the balling process.

Both these furnaces involve the rotatory motion of very heavy weights, and of foundations of ample strength and steadiness. I have made a comparison of the amounts of the weight and movement in the two cases of the cylinder and the carbonating machine. A large cylinder, which can work 4-ton charges of sulphate in  $2\frac{3}{4}$  hours, weighs, when full, 65 tons, and its two rings roll on its four bearing wheels at the rate of  $1\frac{1}{2}$  miles for each charge, or  $6\frac{1}{2}$  miles in 12 hours. A McTear carbonator, working  $5\frac{1}{2}$  tons in six hours, weighs, when full, 29 tons, carried by nine wheels—and the weight is carried along  $5\frac{1}{2}$  miles of single rail for each charge—or 11 miles in 12 hours on one rail. Or it may be taken that each ring of the revolver exerts a pressure of 16 tons per wheel for  $6\frac{1}{2}$  miles, and the single rail of the carbonator carries  $3\frac{1}{4}$  tons per wheel for 11 miles in the same time.

I have said that there is no economy in fuel in the flat circular carbonating furnace. A flat-bedded

circular furnace can never be so economical in fuel as a long one. The flame in crossing the diameter of the bed can never expend its heat, and a large amount must pass away unused. Besides, the radiation from the revolving bottom of such a furnace must be much greater than from a solid bed. The time may come when machinery may be contrived to work a charge from the cold end of a long stationary furnace, to be finished and discharged at the hot end. But even then the difficulty of the stirring and moving machinery being exposed to too much heat would remain to be overcome, not to speak of the adhesiveness of the material requiring actual scraping off the working-bed, which a workman with his tool can accomplish.

We are indebted to Mr. McTear and Mr. Alfred Allhusen for bold attempts to solve the problem of the application of machinery to the decomposition of common salt, and the less enterprising members of the trade will wait with interest the results of their skilful endeavours. The problem is all the more interesting that the one attempts the operation by a continuous and the other by an intermittent method, while they are like each other in the confidence which they have shown in their respective plans and in their energy in overcoming difficulties and in the liberal outlay of capital which they have made in carrying out their designs. My own impression is that it will be found that their plans will not be generally adopted by the members of the trade. The future requirement of the trade is a furnace which will produce hydrochloric acid in a form which can be most easily condensed, and I believe that the less ambitious furnaces at present in use will carry the day in the long run by their superiority in this respect. Besides I consider that the evolution of two-thirds of the acid by the low heat of the pan, and of the rest by the higher heat of the drier, will always be better than using one furnace for the whole operation; and I should expect that by the old method the resulting hydrochloric acid will be more free from sulphuric acid. If I were disposed to experiment on mechanical decomposing, I should not give up the pan which does so well so much of the work, but should supplement it by a rotating hearth, which would do the duty of the present drying bed.

There is little room for controversy as to the economy of machinery in the movement of large quantities of liquids, in making dissolvings and precipitations and other similar operations. But still I believe we have often put up pumps for liquids in cases where a simple steam jet would have answered the purpose equally well, requiring only the opening and shutting of a tap. Again, we have been too much disposed to cover a large space of ground with vessels and apparatus involving an unnecessary expenditure for the erection and maintenance of roofs and an unnecessary amount of horizontal movement of both liquids and solids. A well designed sugar factory of many storeys in height realizes the advantages I suggest, where the raw material is raised to the top storey to begin with, and the successive operations are carried on gradually towards the ground. In the horizontal movement of materials within a manufactory the value of that venerable contrivance the wheelbarrow is very often under-rated. I wish I knew the name of the inventor of the wheelbarrow, for he well deserves the thanks of the many generations which have profited by his ingenuity. Heavy waggons on rails with turn-tables are often used where a dozen wheelbarrows would more easily receive the materials, and one man could wheel away the barrows in succession at less cost for labour, and great saving of outlay in plant. I once saw an elaborate arrangement of iron waggons, with

elevating platforms, high level rails, and turn-table to lay 100 tons a-week of materials in a heap, which I am certain wheelbarrows with an incline could have done more economically. A waggon is confined to its rails, while barrows can range over the whole floor and deposit their loads where required. And, if needed, a steam or hydraulic lift can take both man and barrow up to a higher level of roadway. On well laid level tracks very heavy loads can be easily conveyed in wheelbarrows. I have known 7 or 8 cwt. carried in a well-balanced barrow on a cast-iron floor. And I would here put in a plea for well made barrow tracks—nothing can be more irritating to a workman than to be obliged to use an uneven barrow way, and no trouble should be spared to keep him in good humour in this respect. I have not so much to say for the kindred tool to the barrow, the shovel. It is an economy well worth aiming at to deposit materials in such a way that they will run into the wheelbarrow or waggon or furnace without shovelling.

One of the most ingenious applications of machinery for moving materials which I know is the inclined oven—made of jointed continuous iron plates revolving round a polygonal drum at each end, used in the more recent plants of the Hargreaves sulphate process, and which takes the salt at one point on the ground level and dries and elevates it at the same time, delivering the dried salt at the top of the range of cylinders ready to be dropped into the cylinder that is waiting for it. In a case like this machinery is highly advantageous, and I am only arguing for discrimination in the use of such contrivances. It must be remembered that the fancied saving of manual labour may be accompanied with an expenditure on a higher and more costly class of labour in the shape of repairs to machinery, and it often takes some years before the weak points of new contrivances are discovered, and the ultimate average of repairs can be ascertained. We are in this country a long way from a scarcity of hand labour, and there should be a clear advantage proved before workmen are dispensed with to make way for machinery. And it should be remembered that as labour is displaced by machinery in one department of industry, it so far lessens the motive for attempting the substitution in other departments, by increasing the surplus.

The question of the comparative economy of hand labour and machinery is illustrated in the case of the machine called the steam navvy, and I am indebted to Mr. Messent, the Engineer to the Tyne Commission, for some particulars with which I will trouble you. I need not describe the machine in detail. Its advantages are obvious at first sight, cutting off a ton of stuff from a stiff clay bank and emptying the great bucket into a tipping waggon at every sweep of the arm, making the excavation very rapidly. But it very soon cut away all the material within its reach, and needed to be moved forward. This took 50 men, who had to extend the rails and support them so as to carry 50 tons on the four wheels of the machine, which is very difficult in soft ground—and these men were withdrawn for this purpose, and then went back to their usual work. After a time the repairs became equal in cost to the labour, and at the end of five years there were very few of the original working parts that had not been renewed. There was a balance of advantage to the Commissioners in the more rapid completion of the dock excavations, but the work lost during repairs was a drawback; and Mr. Messent questions the economy of the "machine digger" in cases where the material is so soft and loose that it could be easily shovelled into waggons.

There have been various contrivances recently

introduced into gas works for charging retorts by machinery, and so saving time and gas, and getting rid of a very arduous kind of labour. I have not been at the trouble to make myself acquainted with them so as to enter upon any discussion of their merits, but I shall be glad if my having thus ventured to indicate the conditions of the problem may suggest the discussion of the question in connection with gas manufacture by those who are competent to do so; for I am glad to observe that the gas engineers are fully recognised by our Society as representing an important department of chemical industry. There is room for economies in chemical industry in all its branches—I mean in the way of consumption of fuel. I am not sure that much if any guidance is to be got in this direction from calculating thermal equivalents, for I fancy that theoretical and practical results necessarily widely differ. Take for instance the Hargreave process for sulphate; it is theoretically a heat-producing decomposition, or combustion, that goes on within the cylinders, and yet during the greatest part of the operation the radiation of heat must be corrected by the use of external fire. Theory did not allow for the great length of time, about twenty days, that the salt was in the cylinder, and for the great mass of brick work that required to be kept up in temperature.

By means of the Education Act we can now get young workmen who can be better trusted to make simple observations of temperature, pressure, etc., and it should be more easy than formerly to get them to adjust the draught of a furnace or boiler to the necessity of the case, and so better prevent the waste of heat that arises from pressing an excessive quantity of air through the flues and heating it at the expense of the fuel. I am prepared to expect that a supply of air to furnaces by mechanical means will gradually displace the rough and ready method of a chimney draught, and permit the correct regulation of the air to the amount required for complete combustion. This would be attended with the collateral advantage of diminishing the nuisance from smoke. If the air thus mechanically supplied were heated by some waste heat, the combustion would be much more complete. But I must not forget my own caution as to the necessity of carefully considering whether the cost of obtaining a mechanical draught might not outweigh the advantages.

I have, I am afraid, nothing of interest or novelty to bring before you as to the present position of the alkali manufacture. The inevitable revolution is progressing which will ultimately leave to the ammonia process the production of soda ash, and confine the Leblanc process to the production of caustic soda, crystal soda and bicarbonate. There is still room for improvements in the manufacture of these last-mentioned products. Caustic soda has long been manufactured directly from the tank liquor, and I think crystals will be so also when manufacturers make up their minds to it, and thus the expense of evaporating and carbonating will be saved, and the cost of crystal and bicarbonate proportionately cheapened. Even as regards soda ash, when an article of great density is required the Leblanc ash will be found to have a great advantage over the ammonia soda. There is, I believe, a growing demand for sulphate of soda for glass making, partly in substitution of soda ash.

And this leads me to mention a very great anomaly in the legislation affecting the escape of noxious vapours. By the Alkali Act we are compelled to effect a very nearly complete condensation of all the sulphuric and hydrochloric acid in the manufacture of sulphate of soda. This article is purchased by the glass manufacturer, who decomposes it



with silica in his melting pot and turns out into the air, wholly uncondensed, all the sulphuric acid which the alkali maker is prevented by penalties from allowing to escape in anything beyond the smallest quantities. A similar remark applies to the copper works. They are under no restriction as to the escape of acid vapours, unless they arise from the decomposition of salt, and the injury they may do is naturally laid at the door of the regulated and restricted alkali manufacturer, by the public, who naturally think that that trade must be the most injurious which has attracted the attention of the legislature. While it is true that no practical means of condensation have been devised for glass and copper works, those industries are not even under inspection nor subject to the obligation of adopting the best practical means of preventing escapes which may from time to time be devised. This is to be regretted, as it takes away that stimulus to improvement and watchfulness which has been so useful in the alkali works.

I have been long convinced of what I may call the educative value of the Act. The chambers and decomposing plant are now worked with a minute vigilance that was before unknown. The beautifully exact system of continuous tests has been extensively introduced, so that in the best managed works there is no improvement possible in the yield of sulphuric acid in the chambers, and those who need it know how to obtain the maximum production of hydrochloric acid from the salt in a useful form.

The great desideratum of the alkali trade is an outlet for hydrochloric acid, independently of the production of bleaching powder; and I observe in the Journal of our Society for last month that in Germany a beginning has been made in treating the phosphatic slag from the basic steel process with hydrochloric acid to obtain phosphate of lime for agricultural purposes. It must be the object of the steel maker to make as little of this slag as possible, and to turn it out as rich in phosphorus as possible; and with these richer slags there seems an opening for a large consumption of hydrochloric acid in their treatment. It will be at least an interesting thing to see the iron and alkali industries thus linked together for their common benefit, and for the prosperity of this district, which so much depends on both. In the same Journal another process—also German—is described for the treatment of the phosphatic slags with sulphate of soda for the production of phosphate of soda.

Among the possibilities of the future for the benefit of the alkali trade may be reckoned the recovery of sulphur from the waste. The object of Mr. Gossage of 47 years ago has yet to be realised—and realised without the use of hydrochloric acid. The spirited efforts of Mr. Alexander Chance to carry out the Schaffner and Helbig process, although not yet wholly successful, have been productive of the great benefit of permanently cheapening pyrites in the future; and I think that Mr. Chance's exertions deserve a more adequate recognition from the consumers of pyrites than they have as yet received. The recovery of the sulphur in the waste, not merely as sulphuretted hydrogen to be burned into the chambers, but as brimstone, is not to be despaired of. It is the object of much inventive ingenuity at the present time, if we may judge from the Patent Journals; and if the recovery of the carbonate of lime used in the balling operation can also be secured, the benefit will be all the greater, and the position of the Leblanc process will be fortified by the recovery of all the products which are now wasted.

Although beyond our control, as being a conse-

quence of the policy of foreign governments, I cannot help looking for a stimulus to the chemical as well as other industries from the relaxation or abolition of the protective duties (sometimes prohibitory) levied on our manufactures on the continent of Europe and in America. We are inclined to feel some irritation that this country should be itself suffering from other countries selfishly refusing to follow in that path of free trade in which we set the example, while they are willing to profit by our consistent adherence to our policy. But I am not without hope that wiser and more generous views must before long prevail in those countries; and in the meantime we shall encounter the inevitable changes which science and invention force upon us all the better that we have not the State to rely upon, but only our own industry and energy.

#### DISCUSSION.

Mr. B. S. PROCTOR: It is one of the pleasant duties falling to the lot of a Vice-chairman to move a vote of thanks to the Chairman for his address. It is especially so in the present instance, as we have to thank him for a paper full of valuable practical suggestions, although he has rather surprised me by the conservative tone of his remarks. I could have wished that the duty might have fallen on this occasion on someone else, for I see around me so many practical men who could have spoken with authority on the various points he has raised; but I wish to move a most cordial vote of thanks to the Chairman for his most interesting address.

Mr. ALFRED ALLHUSEN: I have very great pleasure in seconding the motion. I quite agree with Mr. Proctor that the address has a very conservative tone. Two or three points struck me. Mr. Stevenson refers to the carbonating furnace; his remarks as to the effects of the fire were very much to the point. I have been engaged lately in trying to overcome the difficulty by utilising gas-firing, and the results meet the requirements; you have absolute control over the heat and the admission of air. In my opinion gas firing for manufacturing purposes will sooner or later be generally adopted, especially on the Tyne, for although the fuel actually burnt may be weight for weight the same, yet the difference in price between Northumberland steam coal and Durham small coal is quite sufficient to pay for the cost of its conversion into gaseous fuel. Next, as to the decomposition of salt: where large quantities have not to be manipulated I should not advocate machinery, but where you have to decompose 1,000 tons per week machinery is a necessity. You are not only protected so far as the labour difficulty is concerned, but the cost for labour is lower, the consumption of fuel less, and the yield of hydrochloric acid is greater than from the old hand open furnaces. I have listened with great interest to the address, and second the vote of thanks with much pleasure.

Mr. JOHN PATTINSON: I wish also to express my gratification at hearing such an address, which will be likely to raise many points of discussion, and to bring forward a supply of papers during the session. I was glad to hear allusion to the escape of gases from glass works. It has been a subject of great grievance and annoyance to alkali makers that while they were compelled, under heavy penalties, to condense all their acid vapours, a neighbouring glass works was at liberty to send all its acid fumes into the air of the neighbourhood unchecked.

The vote of thanks was then carried unanimously, and acknowledged by the Chairman.

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## Journal and Patent Literature.

### II.—FUEL AND LIGHT.

*Manufacture of Coke, with and without Recovery of By-products.* Dingler's Polyt. Journ. 252 [6], 253 to 256, and partly extracts of papers read before Iron and Steel Institute at Chester (September 1884), by Dr. C. Otto, and by Watson Smith.

E. FRANZEN, of Angleur, Belgium (Ger. Pat. (addition) 24279, March 10, 1883), in the case of shaft ovens (see this Journal III., 101), in order to be able to cut off the

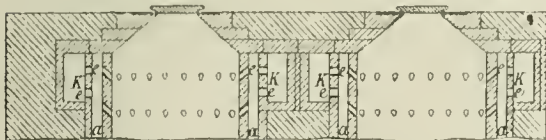


FIG. 1.

heat, to regulate it, and have it more under control, adds in the upper part of the ovens additional mixing-

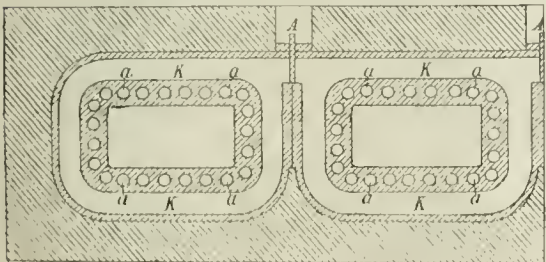


FIG. 2.

flues K (figs. 1 and 2), where mixture with the air takes place. With these extra flues K, all the combustion

channels are connected by the flues *c*. The arrangement is such that when one oven is too cool the damper A can be raised and the fire or gases from a hotter neighbouring oven can be caused to pass into the vertical combustion-flues (side-flues) of the cooler oven.

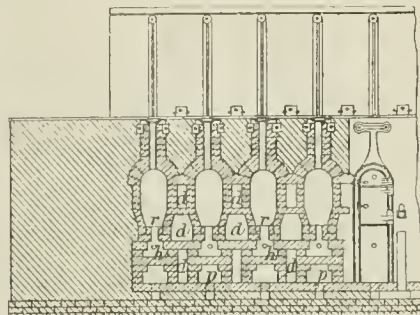


FIG. 3.

In Wintzek's coke-oven (Ger. Pat. (addition) 26131, Jan. 20, 1883), the gases formed in coking pass through the spaces *r* in the sole of the oven into the bottom

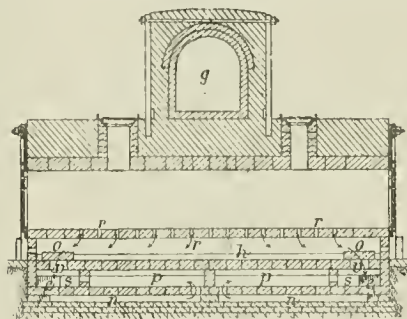


FIG. 4.

channel *h*, and from here, after opening the damper *o* by openings *v*, into the space *s*, then through openings *c* into the horizontal flues *d*, in order to reach the common

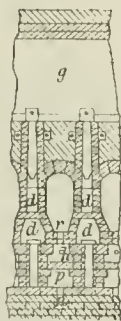


FIG. 5.

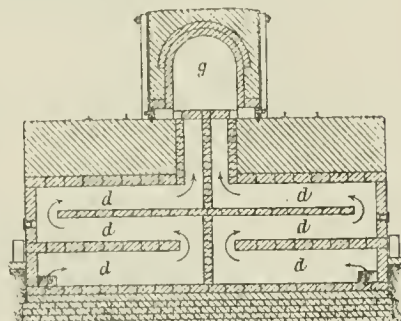


FIG. 6.

reservoir flue *g* (figs. 3 to 6). At the same time air for combustion of the gases is admitted into the space *s*, this air having received a previous heating in the flues *u* and *p*.

C. Otto and Co., of Dahllhausen (Ger. Pat. (addition) 24586, Feb. 23, 1883; see this Journal III. [2], 104), not only pass in the air necessary for combustion of the gases in the side-flues at various points of the bottom or sole-flue, but also the gases themselves are passed in at the different points, and so that their admission may be regulated from without the oven. The gas is admitted



from a pipe or main G (Figs. 7 and 8), passing along the front of the ovens by pipes *c*, just entering the flues, and from the latter by the openings *m* into the bottom

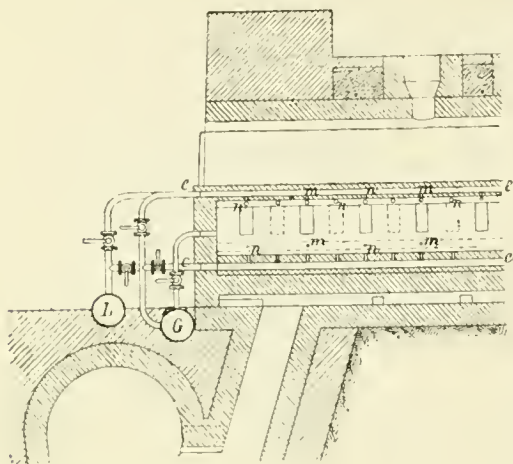


FIG. 7.

flues. The air is also carried in its main L (Luft) in front of the ovens, and enters the separate ovens by the flues *c*, and from these, by the openings *n*, into the bottom or sole-flue. In the gas channel or flue the gas and air mix, and are burnt whilst proceeding upwards in these vertical side-flues.

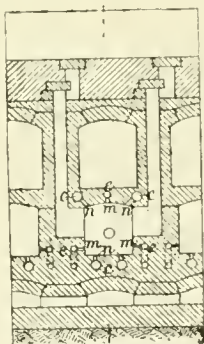


FIG. 8.

With G. Hoffmann's regenerative coke ovens (the Silesian Coal and Coke Works of Gottesberg, Ger. Pat. 25825, May 6, 1883), the gases are drawn through the

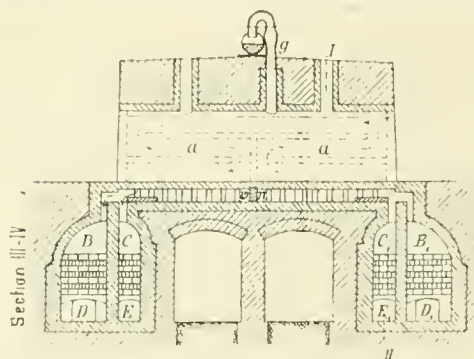


FIG. 9.

pipe *g* to a condensing arrangement, in which the tar and ammonia-water separate, and the residual gas is carried back to the coke ovens, so that according to the

position of the clapper-valve between the ovens and the chimney, this gas may enter either at *E*<sub>1</sub> or *E*. If the valve be placed so that the gas enters at *E*<sub>1</sub> then the air enters by *D*<sub>1</sub>. The gas passes through the regenerator *C*<sub>1</sub>, already very hot from having formed previously the exit-flue for the burnt fire gases, the air being similarly heated by passing through *B*<sub>1</sub>. The recuperated air and gas meet in the sole-flue *n* (Fig. 10) and are partially

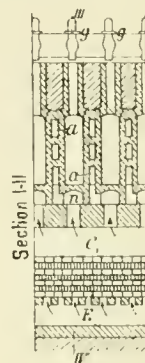


FIG. 10.

burnt, and also farther and completely burnt in the side-flues *a* (Figs. 9 and 10), and in the sole-flue *v*. The hot burnt gases pass off by the regenerators *C* and *B*, and from these through the flues *E* and *D* to the chimney. The pigeon-holed regenerators *C* and *B* are thus heated strongly, and after a certain time the clapper-valve is reversed, and *C* and *B* now become regenerators for the air and gas now respectively caused to enter the combustion-flue from them, whilst *C*<sub>1</sub> and *B*<sub>1</sub> in their turn act jointly as exit-flues for the burnt gas products. The gas, freed from tar and ammonia of course, now enters at *E* and the air at *D*. Recently, however, it was found (Ger. Pat. (addition) 26421, May 7, 1883), that only the air for combustion requires recuperating or heating, and it is better to let the gas enter or be injected into the combustion-flue cold.

[Abstracted from Communications to the Iron and Steel Institute, at Chester, September, 1884, by Dr. C. Otto, of Dahlhausen, and Watson Smith.]—The Siemens regenerator has also been applied by G. Hoffmann, of Gottesberg, to Otto's coke oven, in a manner quite similar to that according to which it was formerly applied to a modified Carvès oven, the air alone for combustion receiving a previous heating. See the following diagrams:—

FIG. 11.

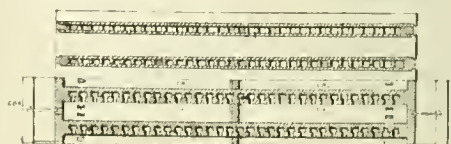
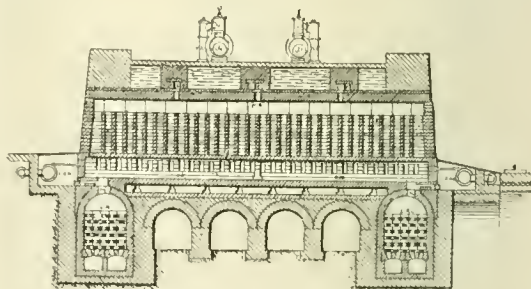
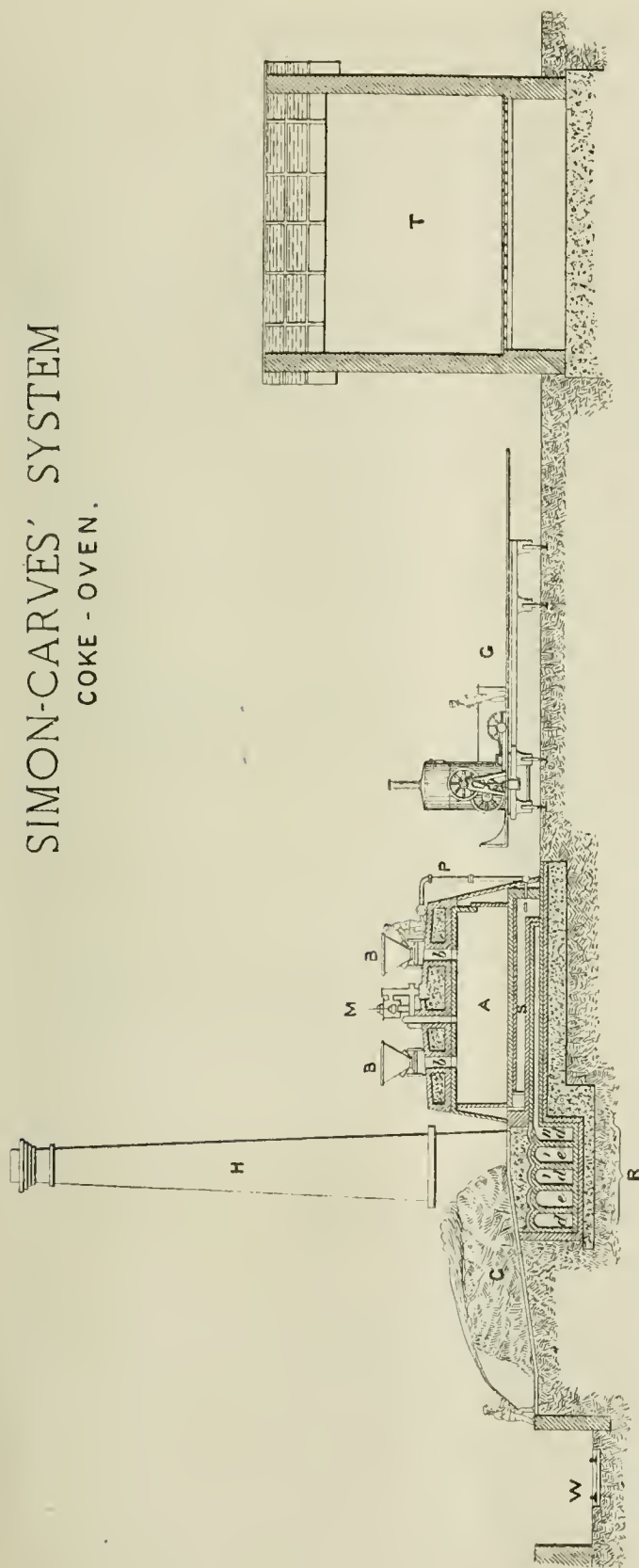


FIG. 12.

Dr. Otto has also devised an elaborate system of condensing apparatus for the recovery of the tar and

# SIMON-CARVÉS' SYSTEM COKE-OVEN.



## LETTERS OF REFERENCE.

- A. Coke oven.  
 BB. Tubs for conveying coal to ovens.  
 G. Steam ram for thrusting Coke out of oven  
 C. Coke, as thrust out of oven, and man applying water-cool it.  
 W. Railway where coke is put into waggons.  
 R. Recuperator, consisting of hot air flues d d d, and smoke flues, e e.  
 S. Main combustion flue.  
 P. Pipe for return gas.  
 M. Main through which gases are drawn by Exhauster.  
 T. Exhauster and pump-house  
 H. Chimney for 25 ovens.

FIG. 13.



ammonia-water. All this is fully described in his paper read before the Iron and Steel Institute, at Chester, in September, 1884. With regard to the products, Otto states that "The yield in ammonia, calculated as sulphate, amounts in Westphalia to 1 per cent. of the weight of dry coal put into the coke oven. This gain corresponds exactly with the contents of ammonia as ascertained by analysis. The quantity of ammonia obtainable from the coal varies in different districts, and even in the same coal district. In the Saarbrücken district the coal yields only 0.7 to 0.8 per cent. of sulphate of ammonia (*i.e.*, 0.7 to 0.8 per cent. on the coal coked), whereas this figure rises in Upper Silesia to 1.1 to 1.7 per cent. The yield of tar at one of Otto's coking works amounted in seven months to an average of 3 per cent. on the dry coal coked. The tar contains less benzene than that produced in gas works, but the amounts of naphthalene and anthracene are equal in amount to those contained in gas tar. The gas produced in the process was analysed after passage through the condensers. It contains a smaller quantity of illuminating constituents than ordinary illuminating gas, though in other respects its composition is similar:—

	Volume per cent. of the Dry Gas.
Benzene vapour .....	0.61
Ethylene .....	1.63
Sulphuretted hydrogen .....	0.43
Carbon dioxide .....	1.41
Carbon monoxide .....	6.49
Hydrogen .....	53.32
Marsh gas .....	36.11
	100.00

The gas returning from the condensers is more than required for heating the ovens, and can be applied for other purposes. It is stated by Otto that by the daily combustion of 2 tons 14 cwt. of coal per oven he can obtain sufficient waste heat from every oven to heat 54 square feet of boiler surface. A heating surface of 54 square feet per coke oven corresponds to an evaporation of 1 lb. of water for every pound of coal coked. These are results obtained with Westphalian coals, and are only comparable with other results with coals of the same quality and district. In Westphalia, with an ordinary coke oven, without extraction of by-products, charged with 5 tons 18 cwt. of coal, 80 square feet of boiler surface are heated, or 1 lb. of water are evaporated by every pound of coal coked. It will be interesting now to compare with this (the process most generally adopted in Germany) the process which is gradually making its way in England, and which has already made its way in France, *viz.*, the Carvés, or Simon-Carvés process. The diagram illustrating the Simon-Carvés coke oven in operation is shown on page 507 (Fig. 13). The amount of ammonia obtained as sulphate from English coal (Crook, by Darlington) was from about 1 to 1.1 per cent. some time ago, when the condensing apparatus was not in order or working well. The tar was then also 2.6 per cent., which would certainly reach 3 per cent. with good condensation. This tar contains less benzene and carbolic acid than gas-retort tar does, though in this respect, according to Mr. Sadler, of Middlesborough, the variations are somewhat considerable. The anthracene and naphthalene, however, amount to nearly three times the quantity found in gas-works tar, the tar is consequently of higher density than gas tar."

[Continuation of Abstract from Dingler's Polyt. Journ.] —O. Ruppert, of Gelsenkirchen, in his coke ovens, with arrangements for recovering by-products (Ger. Pat. 24404, January 17, 1883), has introduced (Figs. 14 and 15) a tubular or flue arrangement *r* into and through the oven, by which through *s* or *f* air can enter, and when heated it passes into the gas-flue at *u*. Fresh and cold air can be admitted at *z*. Instead of the small air shaft *s* being placed on each oven, a large common air shaft can be placed on each pillar-support of a battery of ovens, which is connected suitably with all the vertical flues *s* of the ovens of the battery. The flue *x* also serves to cool the upper part of the oven, and in the case of very long ovens, the channel *v*, which serves to carry

off the gases for condensation. Thus, it is claimed, slight decompositions of the products may be avoided. To avoid mixtures of gas and air beyond control, thus

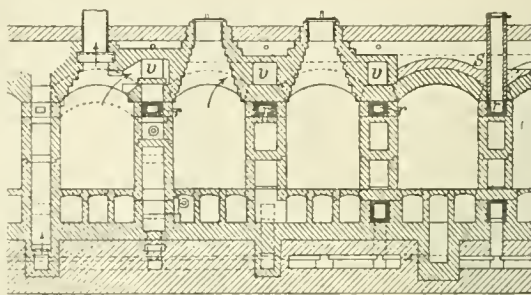


FIG. 14.

leading to combustions at unsuitable points without aim or object, the hot air admission flue *r* is arranged as a separate tube, composed of long pieces united together

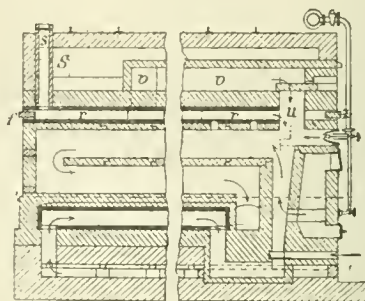


FIG. 15.

and laid between brickwork, so that no hap-hazard mixtures can take place anywhere, and the air only enters into the gas where it is intended it shall.

According to H. Herberz, of Langendreer (Ger. Pat. 25526, June 26, 1883), the gases are drawn off from the

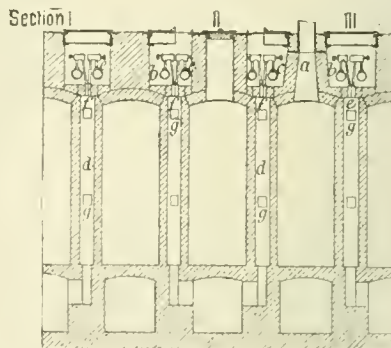


FIG. 16.

vault of the oven at *a* (Figs. 16 and 17), passed to the condensing apparatus, and after separation of tar and ammonia are led through the pipes *c* by *f* into the vertical flues *d* of the ovens. The air necessary for the combustion of the gases is led cold or hot through the pipes *b* by *e* into the single vertical flues, and by cut-off arrangements the admission of air and gas is regulated, these cut-off valves or dampers being fixed on each pipe singly, or on groups of the pipes. In order to have these gas and air mains, and especially the admission apparatus, well protected from wind and weather, as well as to aid in raising the temperature, the gas and air are either to be drawn along by the strong draught of a chimney or to be driven along by pressure. To prevent

the formation of explosive mixtures in the flues, the latter are connected by one or several cross-channels. The inflammation of the gases takes place through these cross-flues when the ovens are set to work. The cooled gases, from which the tar is deposited, are in many cases

preferable to use the gases from the coke ovens for other purposes than for the purpose of heating the same, and then the coke ovens must be heated by specially prepared heating gas.

A. Klönne, of Dortmund (Ger. Pat. 25673, May 2,

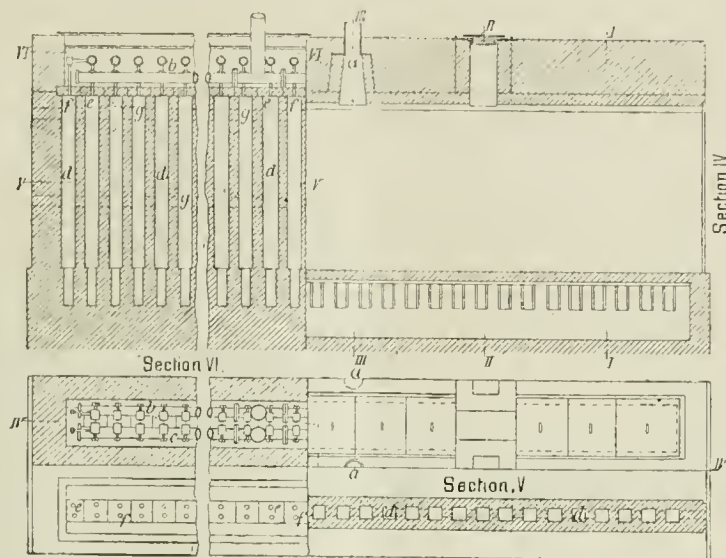


FIG. 17.

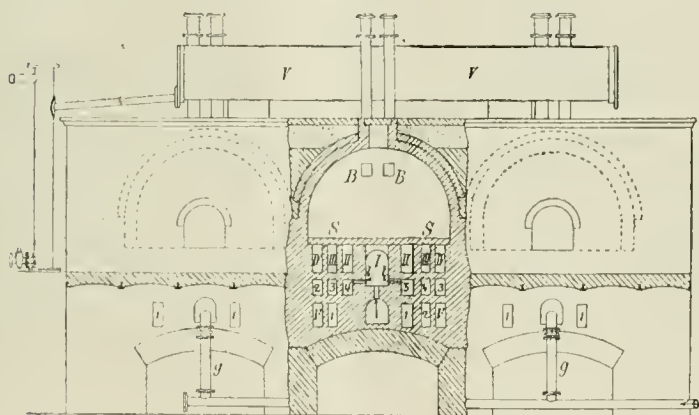


FIG. 18.

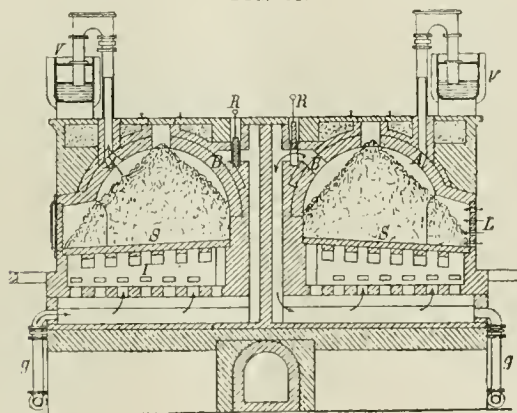


FIG. 19.

not sufficient to heat the coke ovens, and it becomes necessary to assist these gases by the gases of auxiliary gas-producers. Cases can moreover occur in which it is

1883), fills his coke oven (see Figs. 18 and 19) and heats the sole of it, S, with gases freed from tar and ammonia by condensation, these entering at g by a pipe. The



gases pass upwards and enter through the opening A into a receiver V, after closing the second exit B. This is the first period. When as much tar and ammonia as possible are thus driven off, the damper R, connecting the interior of the coke oven with the side-flues and the sole-flues, is opened, and then, for the purpose of the coking process itself, air is admitted into the oven at L by perforations exposed by removing a shield or damper. The coking process now enters upon its second period, the process proceeding from above downwards. The half-burnt gases produced are drawn off by the opening B, and completely burnt by air which has been previously heated in the flues marked 1 to 5 and is admitted into I, and then these burnt gases enter into the same flues 1. to IV. and V. which in the first period had been heated by the illuminating and generator-gases freed from tar and ammonia. The illuminating or generator gases still on hand are either stored or caused to unite with the coke oven heating gases (after condensation), since the presence of the former in the mixture raises the temperature of combustion, and consequently shortens the first or gas period.—W. S.

*Preparation of Coke, Tar, and Ammonia.* Dingl. Polyt. Jour. 252, pp. 283-286.

F. BRUNCK, Mannheim (Ger. Pat. 25499, May 19, 1883), proposes to warm the air and heating gases by employing the smoke-flues alternately for air and gas. He further

Soldenhoff, Louvain, Belgium (Ger. Pat. 25824, March 9, 1883), see Figs. 2, 3 and 4, the coal is submitted to a gradually increasing temperature to obtain a maximum of tar. Each side-wall of the long rectangular chambers, A and B, contains four smaller flues, *c*, *d*, *e* and *n*. The upper flue *c* is connected with the transverse flue G, which can be shut off by the register J, and is connected with the chamber by a number of holes *s*. The channel *c*, provided with an air regulator, has half its top and bottom constructed of perforated brick, in order to pass in air in thin streams. When *d* is connected with G the gases go from *c* to G, then through *d* and *n* to the flue M, which leads to the chimney. If, on the contrary, valve J be closed, then the oven cools, and cold air enters by I in order to cool the channels *d*, *e* and *n*. Air can also be admitted by means of the channels *o* lying in the arch of the oven. Each channel *c* is provided with an iron pipe in front, through which, when the furnace is working cool, the gases are led to the condenser R. The latter consists of an inclined iron pipe, cooled by a water jacket, and provided with a tap at the lower end for drawing off the condensed tar and water, whilst the volatile constituents are drawn through the apparatus V, in which the ammonia is absorbed by water. The non-condensable cold gases pass through the pipe X to the back of the ovens, and thence through the valved tuyere *y* into the channel *d*, where they are burnt. The ovens are charged by means of the shafts S. If the chamber A be shut off from the draught, the gases go from

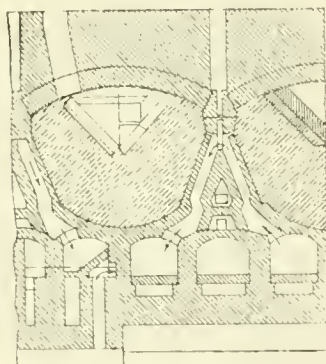


FIG. 1.

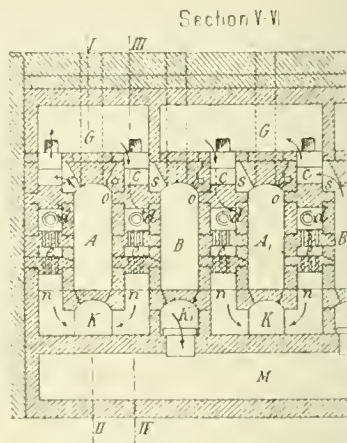


FIG. 2.

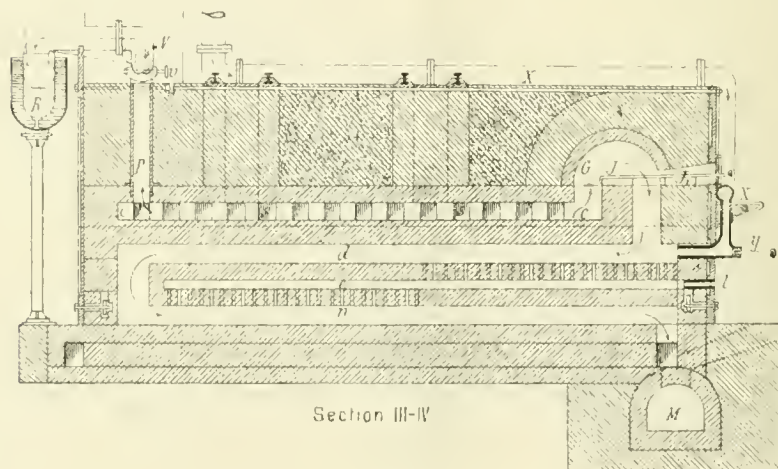


FIG. 3.

recommends a broad dish-shaped oven (Fig. 1), admitting of more sole-flues and a considerable inclination of the walls, which should be thin. In the coke oven of R. de

A through the pipe P to the condenser R, and the oven cools down. If the valve *c* be changed (A connected with R), and A connected with G by means of the

register J,  $A_1$  being shut off from G, then the gases go through the canals  $c, d, e$  and K to M, and the oven A becomes hot, whilst  $A_1$  cools down. The ovens are worked as follows:—A is hot, B and  $A_1$  cold,  $B_1$  and  $A_2$  hot, and  $B_2$  cold. The register J is open, and the gases from A go for the most part through the flues of the left-hand side-wall of  $A_1$ , whilst a small portion goes through to the right-hand side. Both currents unite under A in the channel K, and pass through the flue M to the chimney. The gases from the hot oven  $B_1$  pass into the channels of the fourth side-wall (counted from the left), through the channel K of the oven  $A_1$  and  $K_1$  of the oven  $B_1$  respectively to the flue M.

J. Jameson, Newcastle (Ger. Pat. 24915, Oct. 24, 1882). Furnace for coking (Fig. 5), with central pipe for drawing off products (D), and two smaller pipes for returning

or for subliming. The oven A has a charging door  $f$ , and perforations in the bottom carrying off gases and products to canals connected with pipe  $c$  leading to the condensing apparatus. On one side is a firegrate  $d$ , separated from the oven by a bridge  $e$ , so that hot gases pass over the charge to the flues  $g$ .

H. Hutchinson, London (Eng. Pat. 2843, June 7, 1883), proposes to obtain hard coke and tar rich in benzol, which necessitate rapid heating of the charge, or soft coke and tar rich in paraffin by slower coking, by providing ordinary ovens with canals through which hot air can be passed to accelerate the coking. Fig. 8 shows the oven for the production of heating or illuminating gas, in the centre of which is pipe A for the introduction of superheated steam, and side-flues for admitting heated air.

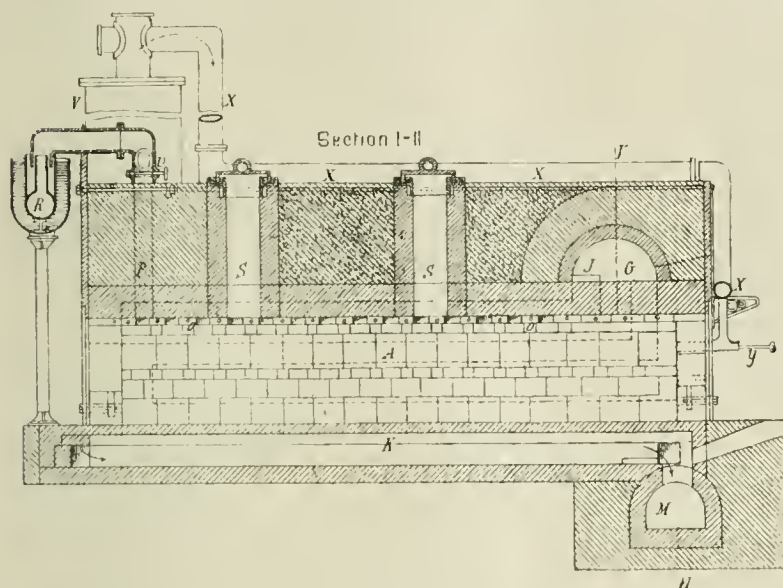


FIG. 4.

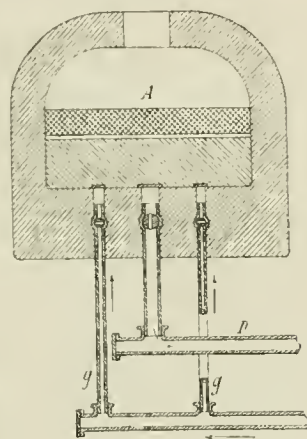


FIG. 5.

certain quantities of the gaseous products, in order to obtain harder coke. Also (Ger. Pat. 25676, June 1, 1883), distillation of non-coking materials, such as shale, small coal, sawdust, &c. (Figs. 6 and 7). When advisable to increase the temperature or to support combustion, the

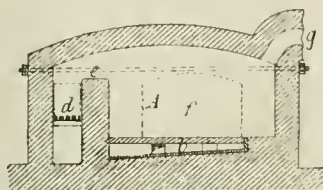


FIG. 6.

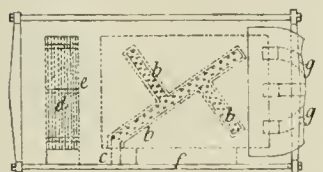


FIG. 7.

material may be mixed with a corresponding amount of coal. The method is also applicable to the extraction of volatile products from ores; also for the production of comparatively pure carbonic anhydride from limestone,

F. Hornig, Dresden (Ger. Pat. 23670, Feb. 18, 1883), describes a scrubber for obtaining tar and ammonia from coking. It consists of a cylindrical vessel A, (Figs. 9 and 10) into which the gas to be washed is passed at B.

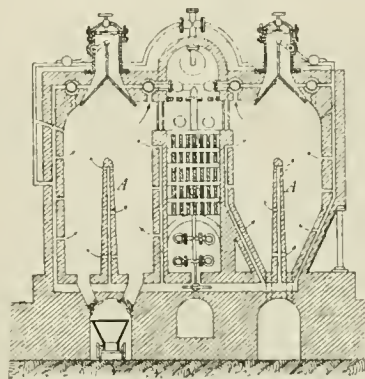


FIG. 8.

The gas passes out by the tube C from near the top of the vessel. Plates D, forming a series of screw surfaces—three, for example in the figure—pass from near the bottom to the top of the scrubber; the gases pass upwards along the canals thus formed, and ammonia water



or pure water is run down the plates to meet the ascending currents of gases. The water is supplied through the tubes E, and is drawn off at F.

FIG. 9.

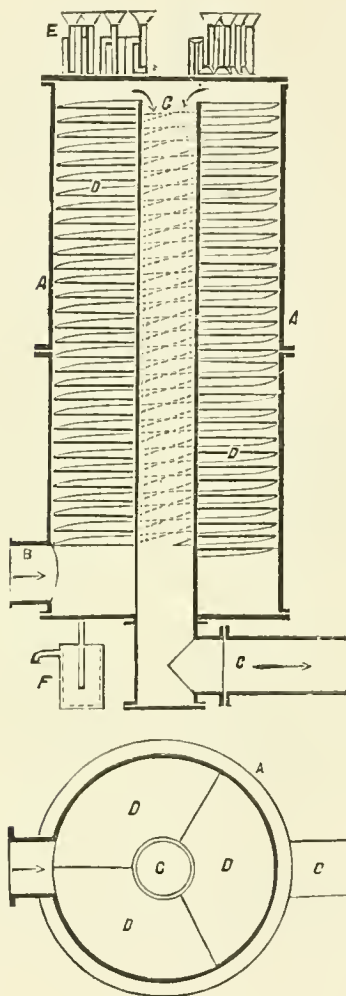
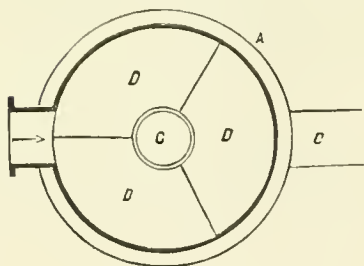


FIG. 10.

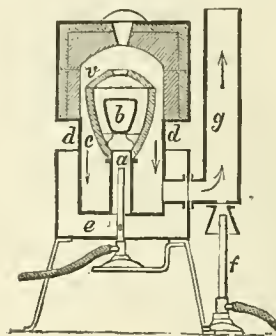


F. P. Dewey (in *Iron*, 1883, vol. xxii., p. 376), has determined the specific gravity of coke by powdering and exhausting under water for from 12 to 24 hours under the bell jar of an air-pump. Twelve samples gave a specific gravity of 1.49 to 1.83, and a porosity of 43 to 61 per cent.—J. T.

*A new small Gas Furnace for Laboratory Purposes.*  
Dingl. Polyt. Journ. 253, 79, 1884.

NOTWITHSTANDING the fact that the temperature of the Bunsen flame is somewhat above 2,000° C., it is very difficult to heat a porcelain crucible to the melting point of zinc, since the losses of heat by radiation and otherwise are so considerable. The furnace here described and illustrated is stated to give very good results; it may be employed for metallurgical work, for the ignition of precipitates, the fusion of silicates, for experiments with glass and porcelain, and may be made use of economically in the melting of small quantities of gold. It will melt silver in 15 minutes, fine gold in 20 minutes, and an alloy of 90 of gold with 10 of platinum in 40 minutes. To some extent this furnace is regenerative in its action.

Cold air, in carefully regulated amount, is admitted to the Bunsen burner *a*, through *c*, and has its temperature raised during its passage by contact with the heated jacket *d*. The combustion takes place within the muffle



*c*, which surrounds the crucible *b*. The combustion gases pass through the cover of *r*, thence by *e* into the chimney *g*. By aid of the second burner *f* the draught of the chimney, and consequently the amount of air drawn through *c*, can be conveniently regulated.—W. D. B.

*On the Recovery of the Ammonia in Coke-oven Gases.*  
Cl. Winkler. Chem. Zeit., 1884, 691.

THE author calculates that if the one-hundredth part of the nitrogen present in the total amount of coal consumed in Germany could be obtained in the form of ammonia, the introduction of Chili saltpetre would no longer be a necessity for manurial purposes. So far, however, as coal is consumed for direct heating, its nitrogen is lost as regards the production of ammonia, and it is only when processes of destructive distillation are had recourse to that the nitrogen is obtained in an available form. The amount of nitrogen obtained as salts of ammonia in the preparation of illuminating gas may be roughly set down at 10,000,000kg.; by how much then might this quantity be increased if the nitrogen in coke oven gases could be made available? To obtain some data for an answer to this question the author made the experiment of coking a known amount of coal of previously ascertained composition, and observing the amount and composition of the coke yielded; the quantity and ultimate composition of the volatile portion were then arrived at by calculation. 100 parts of coal of the following composition—

Carbon .....	58.41
Hydrogen .....	3.75
Oxygen .....	5.99
Nitrogen .....	1.98
Sulphur .....	1.92
Ash .....	10.05
Water .....	18.77
	100.00

yielded on coking : 53.2 parts of coke composed of—

Carbon .....	39.91	equal to	68.3	per cent.
Hydrogen .....	0.26	"	6.9	"
Oxygen .....	1.27	"	21.2	"
Nitrogen .....	0.31	"	28.7	"
Sulphur .....	1.40	"	72.9	"
Ash .....	10.05	"	100.0	"
Water .....		"	0.0	"
	53.20			

and 46.8 parts of volatile matters composed of—

Carbon .....	18.53	equal to	31.7	per cent.
Hydrogen .....	3.49	"	93.1	"
Oxygen .....	4.72	"	78.8	"
Nitrogen .....	0.77	"	71.3	"
Sulphur .....	0.52	"	27.1	"
Ash .....		"	0.0	"
Water .....	18.77	"	100.0	"
	46.80			

Now, proceeding upon Foster's hypothesis that 28 per cent. of the nitrogen in the volatile matters can be obtained as ammonia, and further upon the quite arbitrary assumption that 5 per cent. of the total coal consumed is subjected to processes of destructive distillation, the following results are arrived at: Of the 360,000,000 tons of coal consumed, 5 per cent. of this, or 18,000,000 tons, would be subjected to distillation either for the production of coke or illuminating gas. With an average of  $1\frac{1}{2}$  per cent. of nitrogen this coal would contain 240,000 tons of nitrogen, of which 71.3 per cent., or 171,120 tons would be obtained in the volatile products. Of these 171,120 tons 28.2 per cent., or 42,256 tons, would be yielded in the form of ammonia, whose weight would be 58,596 tons, corresponding to 227,490 tons of sulphate. The cost of coking could not be very large, the chief outlay would be for the item of sulphuric acid, of which 265,140 tons of chamber acid, valued at 10,506,600 marks, would be required. Assuming—and the estimate is an unfavourable one—that the interest on capital, depreciation of plant, and cost of management and labour were to absorb an amount twice as great as that allowed for acid, the cost of a centner of ammonia sulphate would be 6 marks 90 pennings. Thus, even basing calculations upon the present abnormally low price of ammonium sulphate, the country would be the richer by 28,000,000 marks. The author recommends that systematic experiments should be instituted as to the best methods of coking coal. He suggests that the question of the production of ammonia should be kept distinct, and studied apart from that of tar production, so that the factors of each experiment would be either coke and ammonia or coke and tar. The scale on which the experiments are carried out should not be too small, and the author advises that with a view of obtaining accurate information as to the most advantageous temperatures, the retorts should be heated by means of generator gas, and not by the combustion of a portion of their contents.—W. D. B.

*The Production of Gas for Heating Purposes.* II. Bunte.  
D. Ind. Zeit. 1884, 122-23.

THE paper treats of the cost of production and relative heating power of gas. There are three kinds of gas made for this purpose, viz., illuminating, generator, and water gas. Illuminating gas is produced by distilling coal and decomposing the volatile products into combustible gases. Generator gas is produced from gas generators fed with coke, by burning the solid carbon to gaseous CO. Finally, water gas is made by the combustion of red-hot coal in steam. In the manufacture of illuminating and water gas, heat is required and consequently absorbed. Generator gas, on the other hand, is formed with evolution of heat, and the amount of heat given off in the process is not only sufficient to maintain the action, but part escapes with the evolved gases. From the chemical composition of these three kinds of gas, the author calculates that 1 cbm. of illuminating gas with heating power of about 5500 cal. gives out about 5.3 times as much heat as 1 cbm. of generator gas, and twice that of water gas. The illuminating has, therefore, the advantage for heating purposes. It possesses also another advantage, where it is a question of bringing gas to a great distance from a central gas works, for it is evident that a system of pipes of given dimensions can convey about five times as much heating power with illuminating as with generator gas, and about twice as much as with water gas. This fact alone would be sufficient to determine the choice of the kind of gas, because for a continuous production for a large district, not alone the pipes, but the size of the gas-holders and apparatus influence the question of cost. In small districts the case is different, as the concentration of heating power has not the same importance. With regard to the cost of production, that of water gas has not been accurately determined, and it naturally varies in different places. In the case of coal gas the value of the by-products covers largely the cost of production. The author considers that the production of heating gas should be brought into direct

connection with the manufacture of illuminating gas, and be so arranged that one set of pipes serve both purposes. To provide a town or large district with heating gas, it would be necessary to manufacture a cheap illuminating gas, the illuminating hydrocarbons to be used for heating purposes, or a cheap heating gas, which could be mixed with hydrocarbons, or carburised in those places where it is employed for illuminating. In America, water gas for illuminating is used extensively in connection with cheap naphtha oils, whereas in Europe the price of the naphtha prevents its adoption. The incandescent burner discovered by Clamond has been introduced successfully to give water gas illuminating power. Like the Argand lamp the burners can be screwed on to the gas pipes, and so arranged that the gas mixed with air burns from a number of small holes surrounding a conical tube. This tube is covered by a conical basket of fine woven threads of magnesia, which, when heated, glow with a pleasant light of 27 to 30 candle power, or equal to about twice that of an ordinary gas light. As a rule, however, the incandescent burner gives better results with illuminating than with water gas. In spite of vast improvements in the manufacture of water gas, it still appears, that for equal volumes, and therefore without reckoning the heating power, illuminating gas is the cheaper.—J. C.

*Combustion Products of Coal Gas.* A. Vogel, Chem.  
Centr. Bl. 1884, 719.

SINCE coal gas contaminates the air of rooms more largely than candles or oil lamps it is difficult to grow plants in rooms illuminated by gas. This is mainly due to the fact that the combustion products of coal gas contain sulphuric acid, which is injurious to the growth of plants. The author has repeatedly tested window washings of rooms in which gas is used, and has invariably been able to detect the presence of sulphuric acid, whilst the washings from the windows of rooms illuminated by candles or lamps were found to be perfectly free from this acid.—D. B.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*On the Methods of Fractional Distillation used in the Valuation of Chemical Products.* G. Lunge, Chem.  
Zeit. 50, 896.

THE Committee of the German Society of Chemical Industry, appointed to discuss the proposed uniformity in analytical processes for commercial products, have collected information as to the precise methods at present employed for fractional distillation in different works, with a view to prescribing a certain standard method which shall give constant results in the hands of different operators. The author summarises the information given by the representatives of the coal-tar and mineral oil distilling and coal-tar colour industries. With few exceptions glass distilling vessels, and generally fractionating flasks with the thermometer bulb placed in the vapour in the neck, are employed. The English retort method, with the thermometer in the liquid itself, is found at only two works, and is there used exclusively for valuing raw English benzene. Complete dephlegmation is attempted at only one place, where the raw benzene is distilled in a copper flask with Le Bel and Henninger's arrangement (Hempel's glass bead tube is to be preferred on account of simplicity and cheapness); but half the firms use simple fractionating flasks, a few with Wurtz's bulbed necks. Another system, which is to be recommended only for liquids yielding but one valuable ingredient of known boiling point, especially if that point be high, and the substance easily decomposed by continued heating, is that in which the vapour is carried off as fast as it forms, so that liquid once vaporised is never returned into the flask. This is accomplished at two works by placing the horizontal off-take tube close to the bulb of the flask, and by a tube, so sealed through the bottom of the neck that any vapour condensing in the latter is carried by it direct to



the condenser. But complete dephlegmation is to be recommended for mixtures of bodies where several constituents are of value, and whose boiling points differ but slightly. In the apparatus above quoted, greater cooling surface (more bulbs) is to be used for bodies of low than for those of high boiling point. The author uses an arrangement in which the vapour first passes off by an upward sloping tube, thence through a worm surrounded in a tin vessel by a liquid which may be heated to any given temperature, and thence sharply into the usual condensing apparatus. Again, it is of paramount importance that the thermometer shall be accurately graduated. To ensure this a thermometer with a movable scale actuated by a screw may be used; in this case, before making each determination a blank experiment is tried by distilling a chemically pure liquid, whose boiling point is known and approximates to that of the body to be examined, in an apparatus precisely similar to that which is to be employed. Thus the thermometer may be standardised, and there will then moreover be no need to ascertain barometric pressure. As to the exact position of the thermometer bulb in relation to the off-take tube, opinions differ very widely, though it is a matter of considerable importance. The chief points on which uniformity is desirable, are: The material and shape of the distilling vessel, and especially whether dephlegmation is to be employed; in plain flasks, the height of the off-take above the level of the liquid, and the kind of month-piece; the position of the thermometer in the vapour; the length and inclination of the condenser; the rapidity of distillation; the method of collecting; the consideration of other properties and analytical processes, *e.g.*, specific gravity and special methods to be adopted in individual cases.—W. G. M.

*Hydrocarbons and Higher Alcohols in American Petroleum.* G. Lemoine. Bull. Par. 41, 161-166, Feb. 20 (Dec. 28, 1883), Paris Soc. Chim.

THE petroleum employed has the mean density of nonane (0.74 at 15°). By repeatedly fractionating with the bulb apparatus of Lebel and Henninger, the author isolated a certain quantity of nonane. The other portions were again fractionated, and in this way further quantities of nonane and also octane and decane were obtained. The elementary analysis and vapour density determined the nature of the different distillates. The hydrocarbons were then treated with bromine in order to separate non-saturated hydrocarbons, and then distilled in vacuo in such a way that the brominated products remained behind in the retort. The saturated hydrocarbons were again submitted to one or more rectifications in vacuo. In this way the author obtained as principal products octane, nonane and decane. These were treated with chlorine below 65° and the monochloro derivatives prepared, which, by heating in closed tubes to 150° for 24 hours with an alcoholic solution of potassium acetate, gave the corresponding acetic ethers. On saponifying these with potash the alcohols are formed.—I. C.

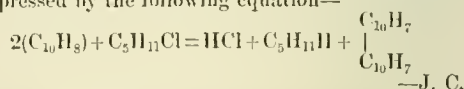
*Furfural.* Antony Guyard. Bull. Par., 41, 289-91.

FURFUROL is always produced by pouring a mixture of equal parts of concentrated sulphuric acid ( $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}$ ) and water, whilst warm, on any substance which consists essentially of a carbohydrate. In this way vapours of furfural can be obtained from starch, glucose, sugar, sawdust, etc. If this be done in a beaker and covered with filter paper soaked in aniline, the presence of furfural can be directly detected by the beautiful red colour, furfuralaniline red being formed. This colouring matter is very unstable and rapidly disappears. Pure cellulose, such as Swedish filter paper, gives very little furfural. It appears, therefore that, in addition to cellulose, the presence of another body is essential to the formation of furfural, the nature of which has, however, not been determined. Bran seems to contain the best proportions of these two substances. Furfural is found in considerable quantities in wood vinegar and gives to it its unpalatable flavour. The author recommends the following method for

separating the furfural. The wood vinegar, separated as effectually as possible from the tar, is shaken for some minutes with benzol (20 to 25 cc. to the litre). Two layers are formed: a benzol layer, which contains the furfural, and an aqueous layer, which gives on one distillation a very palatable table vinegar.—J. C.

*Preparation of Propyl and Amylnaphthalene.* Léon Roux. Bull. Par., 41, 379-382. April 20.

To prepare propylnaphthalene, 200 gr. of naphthalene and 120 gr. propyl bromide are heated in a flask furnished with inverted condenser. As soon as the latter begins to distil, 10 gr. aluminium chloride are added in small portions, and the flask, after each fresh addition, shaken. Hydrobromic acid is given off in considerable quantity, which is received in a previously weighed vessel containing water. The heating is continued until about the theoretical amount of  $\text{HBr}$  is given off. At this point the mass is allowed to cool, and 300 to 400 gr.  $\text{CS}_2$  added, which dissolves, and the solution is heated with water to decompose the chloride of aluminium. The mass is decanted from the carbon bisulphide, dried, and distilled on the water bath. The brown pasty residue is fractionated in vacuo and boils then at 262°–267° C. It forms a compound with picric acid. From the vapour density determination, the author concludes the substance to be a hydrocarbon having the formula of propyl- or isopropylnaphthalene. For the preparation of amylnaphthalene, instead of amyl bromide, amyl chloride is used, as the latter boils at a higher temperature. In exactly the same way as described for the propyl compound, amylnaphthalene is obtained boiling at 288°–292° C. It combines also with picric acid and gives a compound melting at 105°–110°. It is therefore isomeric with that of Paterno (m.p. 140°–141°) and that of Leone (m.p. 85°–90°). In the preparation of the above compounds a quantity of isodinaphthyl is formed. From the fact that Friele and Crafts obtained dinaphthyl by the action of aluminium chloride on naphthalene, the author concludes that the action of the haloid bodies of the fatty series may aid the formation of isodinaphthyl. By dropping amyl chloride into a solution of naphthalene and aluminium chloride, heated to 120°, a strong reaction takes place and a considerable quantity of isodinaphthyl is formed, but no amylnaphthalene. The reaction can be expressed by the following equation—



#### IV.—COLOURING MATTERS AND DYES.

*Colouring Matters from Quinoline.* Walter Spalteholz, Inaug. Dissert., December 22, 1883, Berlin.

THE author tried to prepare the red colouring matter observed by Williams, by treating coal-tar quinoline with amyl iodide and digesting the product with aqueous potash solution, according to Williams; but no insoluble colouring matter could be isolated. The method proposed by Hofmann of conducting the operation in two parts was equally unsuccessful, *i.e.*, by digesting quinoline with amyl iodide on the water bath, and then treating the red crystalline mass obtained with an excess of potash. Methyl and ethylquinoline iodide with potash formed a red colouring matter; but the yield was not more satisfactory. A perfectly pure quinoline was then prepared and treated as above, but no colouring matter was formed. To prepare the colour, equivalent quantities of crude quinoline and ethyl iodide were heated on the water bath, and the solid iodine separated from the alcoholic solution. The latter was further heated on the water bath to complete the reaction, and, after separating the alcohol, the residue treated with about twice its weight of aqueous potash, and heated. On washing with water and extraction with ether, a crystalline product was isolated with a brilliant metallic lustre. To study its composition, which could not be determined from an elementary analysis,

the author prepared quinaldine-ethyl iodide, which he digested with quinoline-ethyl iodide and potash. In this way he obtained a colouring matter identical with that from crude quinoline. An elementary analysis gave the following formula,  $C_{23}H_{25}N_3I + \frac{1}{2} aq.$ , which corresponds to 1 mol. quinoline-ethyl iodide, 1 mol. quinaldine-ethyl iodide, and  $\frac{1}{2}$  mol. water. It therefore appears that the colouring matter from crude quinoline and a mixture of artificial quinoline and quinaldine, are identical, and that pure quinoline from coal-tar and the artificial product are the same body.—J. C.

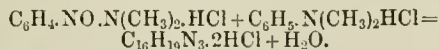
*The Preparation of Blue Colouring Matters.* Dingl. Polyt. Jour. 252, 78, 1884.

ACCORDING to the *Actiengesellschaft für Anilinfabrikation* (Ger. Pat. 25240, May 2nd, 1883), a blue colouring matter containing sulphur is prepared by heating nitroso-dimethylaniline or nitroso-diethylaniline, with an excess of ammonium sulphide. On expelling the ammonium sulphide unacted upon, the colouring matter is obtained as a resinous body, easily separated by filtration from the supernatant liquid.

For the production of a violet colouring matter, A. Bernthsen (Ger. Pat. 25150, May 29, 1883), converts diphenylamine into thiodiphenylamine by coboration of the former with sulphur at 250° or 300°C., and treats the thiodiphenylamine with nitric acid of 40° B. The nitro-compound obtained is a bright yellow powder soluble with difficulty in alcohol and benzene, but easily soluble in glacial acetic acid. On reducing this nitro-compound with stannous chloride, iron, tin, or zinc, a leuco base is obtained, characterised by its yielding a violet colouring matter when saturated with ammonia in presence of an oxidizing agent. The addition of ferric chloride to the solution of the leuco base causes a precipitation of the intensely violet colouring matter, which may be completely thrown down from the solution by the addition of sodium chloride. By recrystallization from boiling water, or by precipitation of its concentrated aqueous solution with hydrochloric acid, the colouring matter is obtained in a state of purity. When dry, it forms a green crystalline powder with metallic lustre, soluble in concentrated sulphuric acid, with a blue colour, which upon the addition of water gradually becomes violet. The aqueous solution of this colouring matter has an intensely violet colour discharged by reducing, but restored by oxidizing agents. Upon heating the dry colouring matter it is decomposed with evolution of hydrogen sulphide. The free base is red and yields with ether a yellowish red solution. Upon animal or animalized fibres this dyestuff is fixed without the aid of a mordant. By treatment of the leuco base with a halogen compound of an alcohol radical, a substituted leuco base is obtained which by oxidation is converted into a colouring matter, the tint of which varies from violet-blue to bluish green according to the alcohol radical introduced. For the preparation of a blue methyl derivative, 4 parts of the violet colouring matter are heated in an autoclave for from 8 to 10 hours at a temperature of 110° to 120° C. with 3 parts of sodium hydrate, 12 parts of methyl alcohol, and 6 parts of methyl iodide. The new base produced is converted into its hydrochloride, and the latter obtained in a convenient form by dissolving it in water and precipitating it as a double salt with chloride of zinc, by the addition of zinc and sodium chlorides.

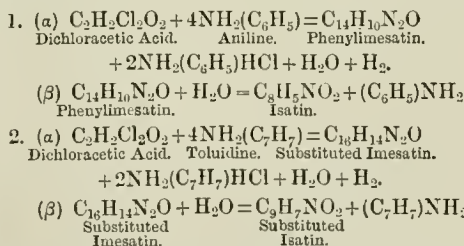
For the preparation of an orange red colouring matter and subsequent conversion of this into a blue dyestuff containing sulphur, R. Möhlau (Ger. Pat. 25828, June 28, 1883) causes 14 kilos. of nitroso-dimethylaniline chloride to react upon 8.5 kilos. dimethylaniline, and 45 kilos. hydrochloric acid of 1.16 sp. gr. at 100° C. The mass froths up and becomes of a brown colour. By the addition of water the colouring matter named *Rubifuscin* is partly, and on neutralization of the free acid present is completely, separated out. Purified by recrystallization it appears in orange red fine needles or in brownish red prisms of orange lustre. This colouring matter is to be regarded as a condensation product of one molecule of nitroso-dimethylaniline chloride, with one molecule of dimethylaniline chloride, one molecule of water being eliminated,

and its formation may be represented by the following equation:—



To obtain the blue colouring matter, the acid aqueous solution of rubifuscin is treated with a reducing agent—hydrogen sulphide has been employed with advantage—and a leuco compound so obtained, which is then oxidized by means of ferric chloride. The blue solution is saturated with sodium chloride, and the colouring matter may then be precipitated with zinc chloride, and brought into a fit condition for further purification. O. Fischer (Ger. Pat. 25827, June 23, 1883) by the condensation of trichlorobenzaldehyde with dimethylaniline or diethylaniline obtains crystalline leuco bases, which are converted by oxidation into bluish green colouring matters. Trichlorobenzaldehyde is obtained by digesting trichlorobenzalchloride (boiling point 280°C.) with 10 times its weight of concentrated sulphuric acid until the chloride is completely dissolved. The solution is poured into water, whereby the aldehyde is caused to separate out in crystalline flakes. On heating 1 part of trichlorobenzaldehyde (melting point 100–111°C.) for several hours upon the water bath with two or three parts of dimethylaniline, in presence of zinc chloride or other condensation reagent, a new leuco base of the composition  $C_{23}H_{23}Cl_3N_2$  is produced. This is soluble with difficulty in water and alcohol, crystallizes from hot alcohol or benzene, and melts at from 128° to 129° C. This leuco base treated in acid solution with an oxidizing agent, as lead peroxide, is converted into a basic colouring matter of the composition  $C_{23}H_{23}Cl_3N_2O$ , whose salts are crystalline and dye the fibre of a bluish green colour.

The preparation of substitution derivatives of isatin, and subsequent conversion of these into substitution derivatives of indigo, forms the subject of a patent by P. J. Meyer (Ger. Pat. 25136, March 2, 1883.) Aniline, toluidine, xylydine, cumidine, naphthylaniline, their alkyl or halogen substitution products, certain diamines—as phenylenediamine, toluylene diamine—are subjected to the action of a di-halogen or di-amido derivative of acetic acid, the mixture being either heated directly or else brought into solution and this subjected to prolonged boiling. The most favourable proportion is 1 mol. of the acid to 4 mols. of the base. The immediate products of the reaction are substituted imesatins, which by the action of strong acids or bases undergo hydration, and are converted into isatin or substitution derivatives of the same. The reactions which take place may be represented by the following equations:—



The di-halogen substitution products of acetone and aldehyde may be employed in place of those of acetic acid. The isatin, or substituted isatin, obtained as above may be converted by ordinary methods into their corresponding chlorides. The reduction of these latter gives rise to the production of indigo, or substitution derivatives of the same.—W. D. B.

*Contributions to a Knowledge of the Colouring Matters of Plants.* Dingl. Polyt. Jour. 252, 183, 1884.

ACCORDING to R. Benedikt (Monatsh. für Chemie, 1884, p. 63), *Istorin*, suspended in glacial acetic acid and treated with nitric acid, dissolves without evolution of gas. From the solution barium hydrate precipitates amorphous by-products and small quantities of a nitro-compound, and in the filtrate therefrom resorcinol acid



may be extracted by agitating with ether. The widely cultivated amaranth (*Helichrysum bracteatum*), the flowers of which are distinguished by their bright yellow colour, and when dried are employed in the arrangement of immortelle wreaths, since the colour is very permanent, contains, according to A. Rosoll, a definite colouring matter, to which the name *helichrysin* has been applied. *Helichrysin* may be extracted from the flowers with difficulty by cold but easily by hot water, spirits of wine, alcohol, ether, acetic, tartaric and oxalic acids, but not by benzene, chloroform and carbon bisulphide. It dyes wool and silk yellow, and forms red and yellow lakes. Alkalis and mineral acids turn *helichrysin* red, and metallic oxides and their salts cause the formation of red precipitates. In alkaline solution *helichrysin* is strongly reduced by both sodium amalgam and sulphurous acid. The constitution of this body appears to be that of a quinone. The abundantly occurring fungus (*Peziza aurantia*), also contains a characteristic yellow colouring matter, to which the name *pezizin* has been applied.—W. D. B.

*The Preparation of Anthraquinone Compounds.* H. Engelsing. Ger. Pat. 26432, August 25, 1883; and Dingl. Polyt. Journ. 252, 183, 1884.

If one part of anthraquinonesulphonic acid be heated with two parts of fuming nitric acid and three to ten parts of sulphuric acid, and, after the frothing has subsided, be maintained at a temperature of 180° to 185° for from 10 to 15 minutes, a product is obtained which, when treated with alcohol, is found to contain a red colouring matter easily soluble, and a violet colouring matter soluble with difficulty in that liquid. The violet colouring matter is soluble in water, and combines with bases to form blue neutral and basic salts. The red colouring matter forms red neutral and blue basic salts. Both these dyestuffs are fixed upon mordanted fibres. By the cautious heating of nitroanthraquinonemono- or di-sulphonic acid to 180° or 185° C., either alone or with one-third or half their amounts of sulphuric acid, the mass swells up and becomes black. The black substance thus formed—an oxy-derivative of anthraquinone—is insoluble in alcohol, ether and glacial acetic acid, soluble in sulphuric acid, and combines with bases to form neutral and basic salts. Both the neutral and basic salts are black, and with the exception of the alkaline compounds quite insoluble in water. The colour is readily fixed upon a mordanted fibre. Methyl and ethyl ethers of this oxy-derivative may readily be obtained by heating the black colouring matter with sulphuric acid and methyl or ethyl alcohol. The ether is purified by sublimation, or by distillation in presence of alcohol or water, when a pure solution is obtained. The methyl and ethyl ethers differ only slightly from one another, are soluble in alcohol, and in the dry state are brown powders with greenish metallic lustre. The neutral salts are soluble in water with a red colour, the basic are blue, and with the exception of the alkali compounds insoluble in water. By prolonged heating of the methyl ether or its barium salt, with an excess of barium hydrate in presence of water, *vanillin* is produced; treated in the same way, the ethyl ether gives rise to the formation of *dioxybenzaldehyde*, which closely resembles *vanillin*. It is also possible to obtain *vanillin* by heating the basic barium salt of the above-described black colouring matter in sealed tubes to 150° to 170° C. with the calculated quantity of methyl sulphonic acid. The application of ethyl sulphonic acid results in the production of *dioxybenzaldehyde*.—W. D. B.

*Separation of Azo-Colouring Materials of Mixed Naphtholsulphonic Acids.* (Dingl. Pol. Journ., 252, p. 440).

ACCORDING to Dahl and Co., Barmen (Germ. Pat. 26 305, May 6th, 1883), instead of separating the naphtholsulphonic acids and preparing azo-dyes from the pure acids, certain azo-compounds can be separated through the different solubilities of their aluminium, calcium, barium, and strontium salts. This is especially applicable

in the case of azo-dyes which are prepared from a mixture of the  $\alpha$ - and  $\beta$ -monosulphonic compounds of  $\beta$ -naphthols with diazonaphthalenemonosulphonic acid and diazoazobenzenemonosulphonic acid, as also in the case of a mixture of the di- and tri-sulphonic compounds of  $\beta$ -naphthol with diazonaphthalenemonosulphonic acid, diazoazobenzene and diazoazobenzenemonosulphonic acid. 50k. colouring matter prepared from  $\alpha$ -diazonaphthalenemonosulphonic acid and the mixed  $\alpha$ - and  $\beta$ -monosulphonates of  $\beta$ -naphthol were, for example, dissolved in 1 cbm. hot water; a solution of aluminium sulphate and ammonia was added, so long as a precipitate still appeared on cooling. After cooling the  $\beta$ -sulphonate of  $\beta$ -naphthols was removed by filtration from the aluminium lake of the dye. From the filtrate the colouring matter of the  $\alpha$ -sulphonate of  $\beta$ -naphthol is precipitated by means of common salt. Similarly, 50k. of the colouring matter prepared from mixed di- and tri-sulphonates of  $\beta$ -naphthol and diazoazobenzenemonosulphonic acid were dissolved in about 1 cbm. hot water and treated with aluminium sulphate and ammonia as long as a precipitate appeared on cooling. After the cooling the  $\beta$ -naphtholdisulphonate was filtered off from the aluminium lake of the colouring matter. If, instead of alumina, an excess of calcium chloride be employed to separate the colouring matters prepared from  $\alpha$ - and  $\beta$ -monosulphonates of  $\beta$ -naphthol and  $\alpha$ -diazonaphthalenemonosulphonic acid, then the calcium salt of the colouring matter of the  $\alpha$ -sulphonate of  $\beta$ -naphthol separates as a crystalline mass on cooling.—J. T.

*Valuation of Indigo.* By C. H. Wolff. Dingl. Polyt. Journ., 253 [6] page 256.

FOR the spectral-analytical valuation of different kinds of indigo, the author shakes .5grm. indigo with 5cc. concentrated sulphuric acid and dilutes the solution to 1 litre. It is then diluted more or less according to the depth of colour, and examined before the spectral-apparatus in a layer of 1cc. thickness. Solutions of different strength naturally give a different coefficient of extinction in the spectrum, which is in direct proportion with the percentage of pure indigotine. If the coefficient of a standard solution of pure indigotine be equal to 100, the following figures were obtained with different specimens:—

	per cent.
Indigotine from Baeyer .....	100.00
" Trommsdorff .....	91.58
" sublimed by Schuchardt .....	83.41
" " Author .....	82.45
" Vierordt .....	81.95
" crystallised by Schuchardt ..	79.08
Indigo, Java .....	72.42
" Bengal fine .....	63.97
" " middling .....	60.47
" " common .....	50.94 (?)
" Guatemala .....	50.70
" Madras .....	23.10
" Manila .....	9.41

—S. H.

*Roccellin.* Emil Roussel. Cent. Blatt., 19, 368.

THIS colouring matter, produced by diazotising naphthyl-aminesulphonic acid and combining it with  $\beta$ -naphthol, is one of the purest of the azo-series. It is employed as a substitute for archil (*roccella tinctoria*) and in certain cases in place of cochineal and madder. *Roccellin* has not yet been fixed on vegetable fibres. In dyeing silk, for which it is used in large quantities, the method of adding soap and acid, and raising in sulphuric acid is usually adopted. For woollen dyeing the author employs the following method. The bath is slightly acidified with hydrochloric acid heated to 50° and the wool left in 15 to 30 minutes, then the *roccellin* is gradually added and the temperature slowly increased during half an hour to 90°, and this temperature is maintained for another half hour. By the addition of chrysoin a colour is obtained which can be used as a good substitute for alizarin red, and the author thinks that, by dyeing military clothing with this substitute, a saving in cost of 50% would be effected. Ink spots can be readily removed from cloth dyed in this way with oxalic acid.—J. C.

*Auramine.* A. Köchlin, Sitzungsber. des Comité der Chimie de Mulhouse, April 1884. Pol. J. 253, 86-87.

THIS yellow colouring matter, manufactured by the Badische Anilin und Soda Fabrik and by Biudschedler and Busch, has rapidly won the favour of dyers. It is the first artificial yellow colour which has been fixed on vegetable fibres with tannin. A steam colour is produced by mixing equal parts of auramine and tartaric acid and six parts of tannin. If the colour be printed on ordinary cloth not prepared with tin, it is fixed, after steaming, with tartar emetic in the usual way, and it will then stand soaping. Auramine can be fixed on wool and gives beautiful shades. Mordanting with oil is, however, more injurious than otherwise. The colour appears to resist well the action of light; but on the other hand it is very sensitive to chlorine. The property by which it can be fixed with tannin enables it to be mixed with other tannin colours for producing yellow shades of malachite green, etc. Auramine can also be employed with mineral mordants like alumina. This new yellow will be a powerful rival to quercitron. A. Poirier, in consequence of the high price of the latter, took the first step in substituting a cheaper yellow, by producing his *jaune solide*, an azo-colour fixed with chrome acetate and which could be mixed with ceruleine, logwood, Couper's grey, etc., for olives and mode colours; but auramine in addition to its fastness gives much more brilliant shades. Flavaniline, used exclusively for silk and wool, is another equally brilliant yellow and is manufactured by Meister Lucius and Brüning at Höchst-on-Main. Flavanilin is best printed with an equal weight of tartaric acid and acetate of magnesia. If it is dyed on cotton previously mordanted with manganese (bistre) a brilliant brown is produced. Those papers printed with a tin salt resist are left white. By oxidising flavaniline on the tissue in the same manner as for aniline black a much lighter nankeen shade is produced than in the case of paratoluidine.—J. C.

*On the Introduction of the Diazo Group into So-called Aromatic Para-compounds.* P. Griess. Berl. Ber. 17, 338.

IN reply to recent papers by Liebermann and St. v. Kostanecki on paraazo-cresol, and Nölting and Witt on orthoamidoazo compounds, the author remarks that long prior to the publication of these papers he pointed out and experimentally proved the incorrectness of the notion that, on introduction into phenols, the diazo group always takes the para position in reference to the phenylhydroxyl group. He further states that he has always combated the notion that by the action of diazo compounds on phenols, only amido and oxyazo compounds are produced if the para position be vacant. Several facts are adduced in support of these statements.—A. S.

*New Colouring Matters for Wines and Liqueurs.* Ztschr. d. allgem. österr. apoth.-ver.; Schweiz. Wochenschr. 22, 143-44.

A FRENCH colouring matter "teinte bordelaise" is, according to author, the thickened syrup of the currant with 4% of alcohol. "Rouge végétale" is another colouring matter which can be distinguished from fuchsin as follows. 100cc. of the suspected wine is freed from alcohol by distillation, the residue acidified and shaken with ether. The ethereal extract is evaporated in a small basin with a piece of white woollen yarn. This turns brick-red in presence of "rouge végétale;" by moistening with ammonia, it changes to violet, which fades and gives a dirty shade. If, instead of ether, acetic ether be used, the wool becomes pink red; with ammonia, violet. "Rouge végétale" can, like fuchsine, be extracted with amyl alcohol, but the solution on addition of ammonia is decolourised if fuchsin be present, and coloured violet in presence of "rouge végétale." By gentle heat and shaking, ammonia takes up the colour from the amyl alcohol and gives a dark brown shade. If acetic acid is added the colour disappears. Under the name of "Safransurrogat" a yellow coal-tar colour has

been employed for several years in colouring liqueurs, confectionery, macaroni, etc., and was considered harmless. Different shades, such as "emerald green" and "carminsurrogat," were produced from it. It is the potassium salt of nitroresol with the addition of 40 per cent. of sal ammoniac to prevent explosion.—J. C.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*The Uses of Asbestos.* B.-und Hüttenm. Ztg. 43, 201.

CANADA produces the purest, and for technical purposes, the best asbestos, the most valuable mineral being the so-called "bostonite" which on account of its long fibrous texture can be used directly for spinning. The Italian kind is contaminated with alumina and silica and cannot be used for engine packing, as it wears the piston. The following are some of the uses of this article. Asbestos yarn is made of thread twisted sixfold and can be used for packing small cylinders and other purposes. It is also made into cord, rope, and woven cloth for filtering acids; asbestos sheets of any thickness, for closing flanges, which, like manholes, have often to be opened. To prevent sticking, the sheets must be covered with oil or graphite. Asbestos colours, for covering paper, carpets, theatre decorations, and also for valuable documents, have been recently introduced. Finally, woven asbestos clothing has been manufactured by the Boston Asbestos Company, to protect the body against fire.—J. C.

*The Production of Asbestos in Italy.* Oesterr. Zeitschr. 31 No. 50; B. u. H. Z. 43, 225.

ASBESTOS is found in the provinces of Sondrio and Piedmont, in the former in about 40 different spots, where it is obtained by blasting the green chloritic and limestone rocks, in which it is embedded in regular layers 7 to 10 and even 50 cm. thick. It is usual to find asbestos with serpentine, where the latter is softer, more homogeneous and massive. In the province of Turin asbestos is obtained from serpentine in 22 different districts, in strata of 1 to 10 cm. thick and 20 m. long. The best comes from Emarese, the most fire proof from Usseglio; that of Campiglia Souna has long fibres, but is often in a state of decomposition. On the whole the mineral from Valtellina has the strongest fibres. The production has recently received an additional impulse from the union of several companies and the yield of the new company alone (United Asbestos Company) is computed at 300 to 1000 tons annually. Wilson in Genoa, and several Turin firms, work up the crude asbestos for pasteboard, rings for steam joints, asbestos boiler covering, asbestos paper for law purposes, theatre decorations and chemical apparatus and for firemen's apparel.—J. C.

*Determination of Wool, Silk, and Cotton in Tissues.* By A. Remont. Chem. News. No. 1294, 123.

THE author takes four portions weighing each 2 grms., puts one of them aside, and boils the remainder for a quarter of an hour in hydrochloric acid at 3 p.c. If after the lapse of this time the liquid is deeply coloured it is decanted, and fresh acid added, in which swatches are boiled for half an hour longer. Finally they are washed in water, and wrung out in a linen cloth. This removes the dressing. The ebullition with dilute acid removes the dyes readily from cotton, less readily from wool, and very imperfectly from silk. Dark coloured silks are most heavily weighted. The iron compounds used for this purpose are completely removed by the process above described, if they do not exceed one-quarter of the total weight of the fibre, which has then a chestnut brown colour. If the weighting is very heavy the material is only partially decolourised. In this case a few threads, after boiling, are incinerated; the weight of the residual ferric oxide is determined and taken into account if it exceeds 5 per cent. One of the boiled swatches is laid aside and the other two are plunged for one or two minutes into a boiling solution of basic zinc chloride at



60° B. (This reagent is prepared, according to Morin, by mixing together 1,000 parts melted zinc chloride, 850 parts of distilled water, and 40 parts of zinc oxide, heating until the last-mentioned article is dissolved.) The swatches are allowed to drain, thrown into water, and thoroughly washed, first with acidulated water and then with pure water. This operation is accelerated by wringing out the swatches each time in a linen cloth. This operation removes the silk. One of the two swatches from which the silk has thus been removed is put aside. The other is gently boiled for a quarter of an hour in 60 to 80cc. soda lye at sp. gr. 1.020. If the boiling is too strong the lye may become so far concentrated as to attack vegetable fibre considerably. The swatch is washed as above, avoiding friction, as the fibres have been rendered brittle by the treatment which they have undergone. All the four swatches are then heated for fifteen minutes in distilled water, pressed, and allowed to lie in the same room in which they had been previously kept. The next day they are weighed. The swatch first laid aside should weigh two grams; a difference of less than 5mgr. may be neglected, but a greater difference must be taken into account. The difference between the weight of this swatch and that of the second represents the dressing; that between the second and third gives the silk. The weight of the fourth swatch represents approximately the vegetable fibre present (cotton, phormium, flax, hemp), but it is somewhat attacked by the soda-lye. In cotton this loss has been found to amount to five per cent. On multiplying the figures obtained by 50 we obtain the percentage of dressing silk and vegetable fibre. The remainder is wool.—J. C.

*Separating Wool from Mixed Rags.* Journ. Soc. Arts, No. 1664.

HEDDEBAULT finds that when rags, of cotton and woollen mixed, are subjected to the action of a jet of superheated steam, under a pressure of five atmospheres, the wool melts and sinks to the bottom of the receptacle, while cotton, linen, and other vegetable fibres remain intact, thus being suitable for the paper manufacture. The liquid mud which contains the wool thus precipitated is then desiccated. The residue, which has received the name of azotine, is completely soluble in water, and is valuable on account of its nitrogen; moreover, its preparation costs nothing, because the increased value of the pulp, free from wool, is sufficient to cover the cost of the process.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Test for Indigo on Coloured Tissues.* E. Prior. Rep. d. Analyt. Ch. No. 13, 193, 1884.

THE cloth tested and supposed to be dyed with indigo and topped with black was cut into small pieces and treated repeatedly with dilute sulphuric acid to destroy the black colouring matter, then with oxalic acid, washed, dried, and the indigo extracted with chloroform in an extraction apparatus. The solution was used for the following reactions: (1) The absorption spectrum showed the characteristic lines for indigo. (2) Part of the solution evaporated to dryness evolved on sublimation violet vapours, which dissolved in sulphuric acid and gave the indigo sulphonic acid reaction. (3) The remainder of the residue left on evaporation was dissolved in sulphuric acid, and white wool dyed in the solution. This dyed sample was further used for the usual indigo tests. The presence of indigo was therefore experimentally proved.—J. C.

*Application of Electrolysis to the Preparation of the Indigo Vat.* F. Goppelsröder. Ding. Poly. Journ. 251, 465.

IN previous publications the author has spoken of the preparation of colouring matters with the help of electrolysis in relation to the application of electrolysis in dyeing

and printing. In this paper he refers specially to the preparation of indigo vats. A large series of experiments has been undertaken on the reduction in the electrolytic way of pure indigotin, indigo, pure aniline black and other colouring matters. In order to prepare the alkaline indigo vat, finely powdered indigo is mixed with a fairly concentrated solution of caustic potash. A suitable copper vessel is half filled with this mixture, and in it is placed an ordinary porous battery cell filled with the same mixture. The copper vessel is connected with the positive electrode, whilst a large piece of sheet platinum, immersed in the porous cell, is attached to the negative electrode of the battery or dynamo. The whole is gently warmed and the current allowed to pass for three or four hours. Copious evolution of hydrogen and formation of froth are manifested in the copper vessel and the characteristic smell of the indigo vat is soon noticeable. The indigo blue is converted into white, which dissolves in the alkaline solution, giving a yellowish green fluid, which, in contact with the air, soon becomes covered with a film of indigo blue. Woollen materials can be dyed with this vat just as well as with any of the ordinarily used ones. At the positive pole only a slight action takes place, a description of which is reserved. Instead of waiting for complete preparation of the vat, woollen materials may be immersed in it soon after the current has been made, and thus the two operations conducted simultaneously. The hydrogenation of the vat takes place even in the cold, and the operation is so simple that it may be readily carried out on a large scale. As previously shown by the author, it is possible, by electrolytic means, to produce patterns by discharge on the dyed fabric. In the case of indigo a solution of nitre acidified with sulphuric acid is used. A platinum plate resting on a slab of caoutchouc or glass forms the negative electrode, and on this the above solution is placed. The die of gold or platinum or the planished block forming the positive electrode is then laid on the fabric, or if preferable the die may be placed under a slab of lead which is connected with the positive electrode. On passing the current white patterns are produced. If the fabric be used 8 or 16 fold, so as to yield to pressure, very fine impressions are produced. This is applicable not only to the production of discharge patterns on goods dyed with indigo, but also for the production of the most varied colours (aniline black, naphthylamine violet, etc.) on white materials by the simultaneous discharge of indigo blue and Turkey red. Instead of using the acidified solution of nitre, the author has also used a solution of ammonium chloride or sodium chloride and citric, or a mixture of citric and tartaric acids, as previously described by him for electrolytically discharging Turkey red.—A. S.

*Employment of Electrolysis for making up the Indigo Vat.* By Prof. F. Goppelsröder. Dingl. Polyt. Journ. 253 [6], 245-252.

REFERRING to his previous investigations on the same subject (Dingl. Polyt. Journ. 251, 465), the author has continued his experiments, and repeated those which were in opposition to the views of Prof. Wartha (Chem. Zeit. 1884, No. 25, and Centralblatt f. Textilindustrie, 1884, 521). Indigo of different origin, mixed with solutions of caustic alkalis or milk of lime, was subjected at different temperatures to the action of the galvanic current. The reduction of indigo-blue to indigo-white is very incomplete at common temperatures; it is much weaker than that obtained if heat is employed. To bring about a quick reduction, the mixture must, indeed, be raised to a boil. A continued action of the galvanic current—in the cold as well as in the heat—has the effect of exceeding the reduction to indigo-white, and thus destroying the vat. The author is at present engaged with experiments in adding a substance to indigo-white which may arrest the reduction at a certain stage. He is also going to make quantitative determinations on the extent of the reduction. At present his results are only of scientific interest, and far from being of practical value. He also points out that, in the usual way of preparing the indigo vat, it

not unfrequently occurs that the reduction to indigo-white is exceeded. He then adds the details of twenty-one experiments which cannot be abstracted, but must be read *in extenso*.—S. H.

*On the formation of Indigo-white on Cloth by the Galvanic Current as a means of Dyeing Indigo-blue.* By Prof. F. Goppelsroeder. Dingl. Polyt. Journ. 253 [9], 351-355.

IN his previous experiments the author only prepared an indigo vat by the action of the galvanic current on a mixture of indigo and alkali solutions. The cloth was dyed by suspending it in the vat while the current was passing through it. He now reduces the indigotin on the fibre itself, so that the cloth, after exposure to the air, is dyed permanently blue. Extremely fine powdered indigo and solutions of caustic alkalis, or milk of lime, are mixed in the usual proportions, and cloth steeped in the mixture. It is then put between two metal plates, which serve as electrodes. As soon as the current passes through the tissue, the indigo-blue is reduced to white. On the large scale, this mode of proceeding can perhaps be imitated by passing prepared cloth between two metal rollers, acting as electrodes. On certain spots white discharges, or other colours, could be produced; or reserves for white or other colours could be printed on certain spots before the cloth is subjected to the electro-chemical treatment. Next follow the details of forty experiments.—S. H.

*Preparation of Wood Mordants in the Solid Form, for Staining.* L. E. Andés. Chem. Centr. B. 1884, 702.

*Oakwood Mordant.*—5 grms. "kasselerbraun" are boiled for one hour with 0.50gm. potash, and 10 grms. rainwater. The mass is then passed through a cloth filter, the clear solution evaporated to a syrupy consistence, and allowed to solidify in shallow iron boxes. It is then ground to a coarse powder.

*Pale Oakwood Mordant.*—3 kilos catechu are boiled with 1 kilo of rainwater. The mixture is filtered through cotton, and the clear solution evaporated to a syrupy mass. It is then treated with a solution of 250 grms. potassium dichromate in 2 kilos water, evaporated and dried in the above described manner.

*Nutwood Mordant.*—3 grms. dark "kasselerbraun" are boiled with 0.3 grms. potash and 7 grms. water, and passed through cotton. The solution is treated with 2.5 kilos. extract of logwood, and evaporated to a syrupy consistence. The drying is effected in iron boxes as before.

*Rosewood Mordant.*—5 grms. Brazil-wood are dissolved in boiling water, 1 kilo "kasselerbraun" is simultaneously boiled with 3 kilos water, and 0.1 grms. potash, and filtered. The solutions are then mixed together and dried as before.

*Mahogany-wood Mordant.*—3 kilos Brazil-wood are boiled with 0.25 kilos potash and 3 kilos water, treated with 150 grms. of eosin, and evaporated.

*Satinwood Mordant.*—3 kilos extract of fustic are boiled with 7 kilos. rainwater, filtered, evaporated, treated with a solution of 100 grms. potash in 350 grms. rainwater, and dried.

*Ebony-wood Mordant.*—5 kilos. extract of logwood are boiled in 11 kilos rainwater, and filtered. The solution is treated with 300 grms. iron nitrate, and evaporated to dryness.

It is stated that hitherto the manufacture of wood mordants in a solid form has been carried on in England only.—D. B.

## VII.—ACIDS, ALKALIS, SALTS.

*The Manufacture of Sulphuric Acid from Pyrites in America.* G. Lunge (Dingl. Pol. Journ. 252, 293-295).

THE pyrites of the Sulphur Mines Co. of Virginia, according to Dr. A. Voelcker, contain:—

Sulphur .....	48.02
Iron .....	42.01
Iron oxide .....	1.33
Sulphuric anhydride .....	0.44
Silica .....	7.60
Copper .....	0.00
Arsenic .....	0.00

Other analyses show the absence of arsenic and an amount of sulphur of 46.4 to 50 per cent. The ore is found near to the surface, and being frequently in the form of sand is very suitable for step burners. The mine of the Davis Company at Charlemont, which supplies four works, averages 48.5 per cent. sulphur, with no trace of arsenic, antimony, or cobalt, and little or no zinc, lead, or calcium, and less than 3 per cent. of silica. It has a granular structure, is easily broken by hand, and gives a good gas; the residue contains not more than 3 per cent. sulphur.—J. T.

*Separation of Arsenic from Saline Solutions.* F. C. Glaser, Berlin. Ger. Pat. 26632, March 25th, 1883. Dingl. Pol. Journ., 252, p. 304.

THE concentrated neutral saline solution is treated with freshly-prepared stannic oxide, antimonious oxide, antimonious oxide, or lead dioxide, well agitated and allowed to stand 12 hours. The solution tested with Marsh's apparatus should give no trace of arsenic. The deposit is then filtered off, and its arsenic removed by treatment with dilute sulphuric or nitric acid; the residue after repeated washing serves again. The requisite amount of stannic oxide, antimonious oxide, antimonious oxide, or lead dioxide depends on the amount of arsenic present, but about 4 per cent. is usually sufficient.—J. T.

*Improvements in the Construction of Lime-Kilns.* Dingl. Polyt. Journ. 253, 80, 1884.

E. A. SCHOTT (Ger. Pat. 24654, April 28, 1883) builds up the interior of his kiln in the manner here described,

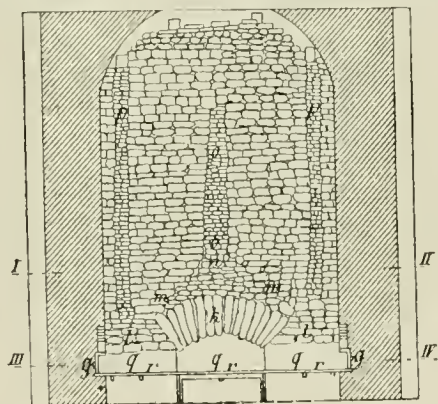
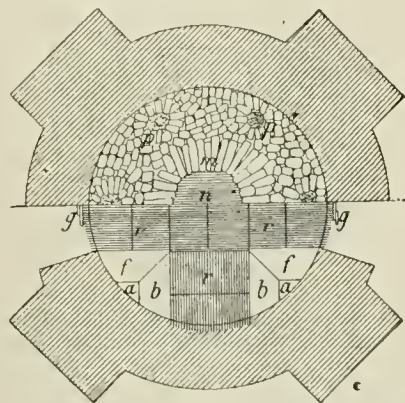


FIG. 1.



Section III-IV

FIG. 2.

and illustrated in figs. 1 and 2. As shown in the ground-plan, four three-sided prisms *a* spring from the interior and are built up to a height of about 50cm. above the



hearth *r*. Upon these prismatic projections the supports *bb* and *ff* rest; between the former *bb* the arch *k* of keyed-shaped pieces of limestone is built up; between the latter *ff* the arches *ll* are arranged at right angles to *k*. In order to contract the centre space *n* long pieces of limestone *m* are so built up that somewhere about *o* this space is only about 20cm. in diameter. This centre channel *o* is carefully covered in with limestone that the combustion gases may be dispersed through the kiln. Six or more flues *p* are also arranged at different points near the kiln walls, and are surmounted by pots; these flues *p* are in connection with the combustion space *q*. Upon the arches *l, k, l*, the mixture of limestone and fuel is built up. On inflaming the fuel upon the hearth *r* and opening the doors *g*, the combustion gases generated in *q* collect under the dome *l, k, l*, pass through *n* and *o*, and thence find their way to all points of the charge.

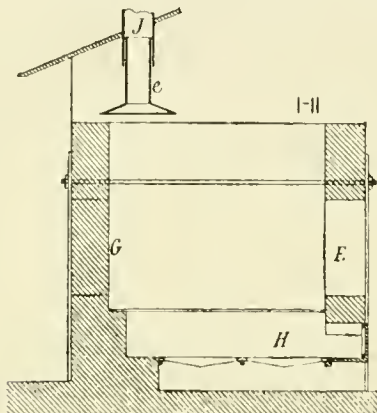


FIG. 3.

If it be desired to burn cement, tile-ware, pipes or pottery, the dome *l, k, l* is built up in the same manner as above, and may be composed either of limestone or fire-clay. The doors for the introduction of the charge are disposed at different points of the circumference to prevent a weakening of the kiln walls. The furnace devised by L. T. Lescigneur (Ger. Pat. 27055, September 23, 1883),

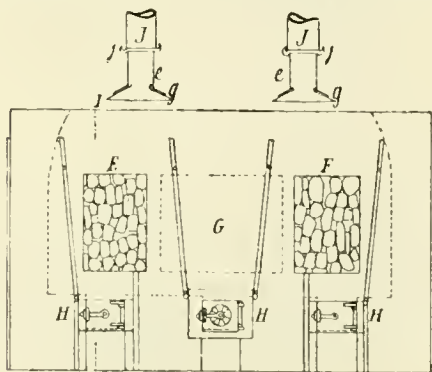


FIG. 4.

Figs. 3 and 4, has openings *E* and *F* in the front wall and *C* at the back, for the introduction and withdrawal of the charge, and it is claimed that by this arrangement easy access may be had to any part. The three firing chambers *II* are covered in with a grid, upon which the charge is laid. The exit pipes *J* which serve for the withdrawal of the products of combustion have fitted at their lower end the sliding tube *e* expanded below to a funnel shape. By the aid of the catches *g* and *j*, *e* may be maintained at such a height that no hindrance is experienced in filling or emptying the furnace.—W. D. B.

*On a Sublimate deposited in the Gas Delivery Tube of a Limekiln.* W. Gintl, *Berichte der österr. Chem. Gesellschaft*, 1884, p. 6.

DURING the more energetic periods of the working of a limekiln in a sugar works, a sublimate was observed to be deposited in the carbonic acid delivery tube, which it more or less obstructed. The composition of this sublimate was found to be—

Calcium carbonate .....	31.97
Calcium oxide .....	18.61
Calcium sulphate .....	17.08
Calcium sulphite .....	1.65
Calcium silicate .....	6.10
Potassium carbonate .....	1.12
Sodium carbonate .....	16.78
Lithium carbonate .....	0.69
Calcium chloride .....	0.49
Alumina and ferric oxide .....	2.54
Manganese, magnesia and the like .....	Traces.

Gintl believes that the above metals were originally sublimed as chlorides, which were decomposed by the hot gases to form the compounds enumerated.—W. D. B.

*The Preparation of Strontium Hydrate.* Dingl. Polyt. Journ. 253, 82, 1884.

ACCORDING to the specification of the Dessauer Actien-Zuckerraffinerie, Dessau (Ger. Pat. Addition 26800, July 24, 1883), the hearth of their gas furnace is so modified

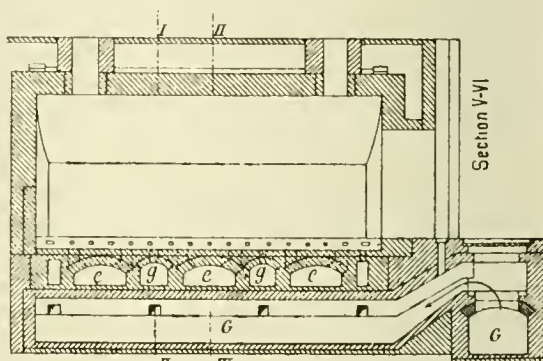


FIG. 1.

that as shown in figs. 1, 2 and 3, the hot air enters by *c* and *a*, the gas by *G, g, c*; the mixture of the gas and air, therefore, takes place in the chambers. Since combustion

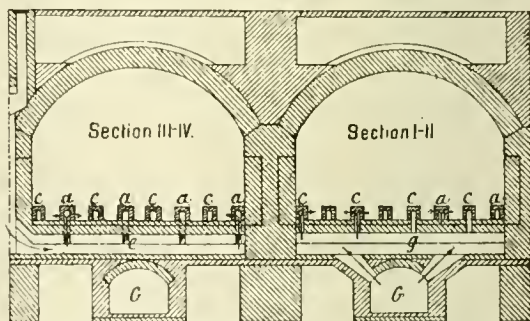


FIG. 2.

commences in the chambers, the fullest advantage is obtained of the high temperature, the waste gases rapidly pass off, whilst at the same time the furnace hearth is but slightly attacked. R. Gionzyenski (Ger. Pat. Addition 27157, June 30, 1883) proposes to cover the steam pipe *e* with a built-up sheath provided with a large

number of jets *c* (see figs. 4 and 5). As these jets incline downwards from within, they are to some considerable extent prevented from becoming choked up by the

hydrate (Ger. Pat. 27159, August 31, 1883), and for the production of sulphide of zinc and strontium hydrate

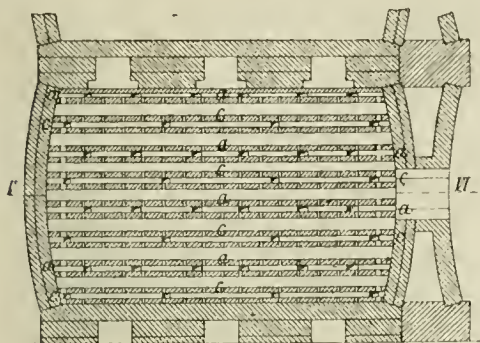


FIG. 3.

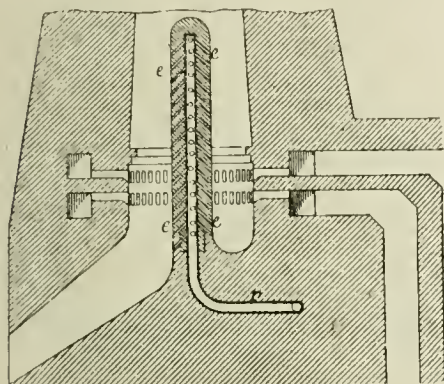


FIG. 4.

material being treated in the furnace. The processes patented by C. F. Claus for the preparation of strontium

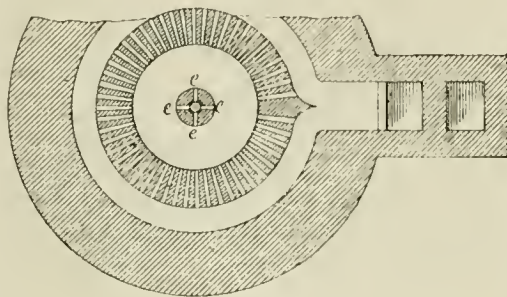


FIG. 5.

(Ger. Pat. 26418, March 2, 1883) have already been described in the *Journal of the Society of Chemical Industry*, p. 476, 1882.—W. D. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, CEMENTS.

*Cement, its Application and Testing.* Dingl. Pol. Journ. 252, pp. 426-431.

E. BÖHME concludes from numerous experiments that as regards tenacity, an unmixed cement always has the advantage over a mixed cement; and with respect to resistance to pressure or crushing, a mixed cement again has a lower value than that possessed by an unmixed one. *Tensile strength* is always less for mixed than for unmixed cement. Tabular results given by R. Dyckerhoff confirm the above. For admixture Dyckerhoff employed crushed slag, fine sand, limestone, and calcium hydrate.

R. Fresenius took 12 samples of unmixed cement of English, German, and French manufacture, along with three sorts of hydraulic lime, three sorts of weathered slag powder, and three sorts of ground slag. The results obtained by him are given in the following table :—

Description.	I. Specific Gravity.	II. Loss on Ignition.	III. Alkalinity of Aqueous Solution from 0.5g. in Deci-normal Acid.	IV. 1g. takes of Normal-Acid	V. 1g. reduces of Potassium Permanganate	VI. 3g. absorb of Carbonic Anhydride
PORTLAND CEMENT			cc.	cc.	mg.	mg.
A	3.155	1.58	6.25	20.71	0.79	1.4
B	3.125	2.59	1.62	21.50	2.38	1.6
C	3.155	2.11	4.50	20.28	0.93	1.8
D	3.144	1.98	5.10	21.67	1.12	1.0
E	3.141	1.25	6.12	19.60	0.98	1.6
F	3.134	2.04	4.95	20.72	1.21	1.1
G	3.144	0.71	4.30	22.20	0.89	0.0
H	3.125	1.11	4.29	20.30	1.07	0.7
J	3.134	1.00	4.00	19.40	2.01	0.0
K	3.114	0.34	4.21	20.70	0.98	0.0
L	3.154	1.49	4.60	18.80	2.80	0.3
M	3.125	1.25	5.50	20.70	2.33	0.0
HYDRAULIC LIME						
A	2.411	18.26	20.23	21.35	1.10	27.8
B	2.551	17.82	22.73	26.80	0.93	31.3
C	2.520	19.60	19.72	19.96	0.98	47.7
WEATHERED SLAG						
A	3.012	0.76	0.91	11.19	74.60	3.6
B	3.003	1.92	0.70	13.67	60.67	3.5
C	2.967	1.11	1.00	9.70	41.31	2.9
GROUND SLAG						
I.	3.003	0.32	0.31	3.60	61.10	2.4
II.	2.873	0.43	0.11	8.20	73.27	2.2



If a cement gives values which fall without the bounds of the above table, adulteration may be inferred. To test the sharpness of the indications, cements mixed with 10 per cent. of the various substances were examined. In all cases the results were sufficiently conclusive. R. Weber, employing a magnifying power of 50-60, after adding acetic acid, found the cement particles to be clearly crystalline, whilst those of slag-powder were amorphous. The method, however, fails with particles which pass a sieve of 5,000 meshes. Dr. Heintzel proposes a fusion test for manganese, as a means of detecting the admixture of slag-powder; since fine cement contains a minimum of manganese whilst slag is comparatively rich in it. The German Cement-makers' Association have decided that the pressure test only can be considered as the standard test for hydraulic binding materials, whilst the pulling test only serves as a qualitative test of the uniformity of the material.—J. T.

*On the Decomposition of Cement by Water.* H. Le Chatelier. Chem. Zeit. 38, 678, 1884.

By the well-known fact that hydrated cements treated with a large excess of water part with some of their lime, it has, up to the present, been considered proved that this lime existed in the cement in an uncombined state. It appears, however, from the author's experiments that lime goes into solution even when it occurs in the cement only in the combined state, and hence he infers that water has a decomposing action upon some of the constituents of the cement. The free lime may, however, be estimated by solution if only a very small quantity of water is employed for the extraction, and this renewed only when it is saturated with lime, that is, when it contains 1·3 grms. per litre. Under such circumstances none of the lime compounds suffer dissociation. It may here be noted that cements which set slowly always contain a large amount of free lime, from which, however, quick-setting cements are almost free. On subjecting a hydrated cement to the progressive action of water, all the lime compounds which have formed during the setting are successively decomposed, and the decomposition of each compound is indicated by the amount of lime in the solution remaining stationary until such compound is completely dissociated. By observing the stationary maximum amounts of lime which a litre of water can bring into solution from different known artificially prepared compounds of lime, and comparing with these the stationary quantities of lime brought into solution from cement by treatment with successive quantities of water, the author considers it possible to arrive at the compositions and quantities of the different compounds occurring in a hydrated cement. In this way he considers the occurrence of the following compounds to be proved: Compounds—maximum stationary amount of lime in one litre of solution—

Ca(OH) <sub>2</sub> .....	1·3 grm.
Fe <sub>2</sub> O <sub>3</sub> , 1CaO, 12H <sub>2</sub> O .....	0·6 "
Al <sub>2</sub> O <sub>3</sub> , 1CaO, 12H <sub>2</sub> O .....	0·2 "
SiO <sub>2</sub> , CaO, 3H <sub>2</sub> O .....	0·05 "

W. D. B.

## X.—METALLURGY, MINING, ETC.

*Swedish Iron Ore.* P. V. Schwarze. Stahl und Eisen, 1884, p. 307.

THE author discusses the expediency of importing iron ore into Germany from Sweden. The most important sources are in the provinces of Örebro and Norrbotten, and the following analyses show the quality of the ore obtained at Granges, in the province of Örebro:—

Silicic Acid .....	1·85	3·61	2·63
Alumina .....	1·02	1·87	1·07
Ferric Oxide .....	70·88	77·41	65·61
Ferrous Oxide .....	22·81	9·18	20·34
Lime .....	1·91	4·10	6·11
Magnesia .....	0·65	1·14	0·65
Phosphoric Acid ..	1·200	2·338	1·411
Sulphur .....	0·004	Traces	0·011
Manganous Oxide ..	0·11	0·09	0·45
	100·194	99·798	100·982

The author considers that the importation of this ore into Upper Silesia and Moravia might be advantageously carried on. A sample of Luossavaara ore gave the following results on analysis:—

Ferric and Ferrous Oxides .. ..	97·65
Manganese .. ..	0·00
Alumina .. ..	0·39
Magnesia .. ..	0·11
Copper .. ..	Traces
Arsenic .. ..	Traces
Phosphoric Acid .. ..	0·05
Sulphur .. ..	0·00
Residue .. ..	1·60
Water .. ..	0·20
Titanic Acid .. ..	0·00

100·00

W. D. B.

*On Manganese Determinations in Iron Works Laboratories.* Professor A. Ledebur. Chem. Zeit. 51, 910, 52, 927 and 54, 963.

THE introduction of certain processes, such as Bessemer's, Siemens', etc., has created a demand for manganese, which will probably never be produced on a large scale, since it fuses only at a very high temperature and is at that heat very volatile. Rich manganiferous pig-iron (sometimes even containing over 80% Mn) may be produced in the blast furnace; and the greater the proportion of iron and carbon the lower will be the fusing point, so that the cost of ferro-manganese increases in geometrical progression with the percentage of manganese present. Hence an accurate method for manganese determination is needed, and it is essential that the process shall not demand too much time or labour. At present there is no process used universally in iron-works laboratories, and the author in this paper gives the results of experiments made for the comparison of certain suitable methods, viz., the volumetric processes of Volhard, Pattinson and Hampe. The same sample of ferro-manganese, submitted first to accurate gravimetric analysis, was used in every case. For gravimetric experiments, 1 gm. of the alloy was dissolved in nitric acid, evaporated with some ammonium nitrate to dryness, taken up with hydrochloric acid, diluted to  $\frac{3}{4}$  litre, neutralised with ammonium chloride and carbonate till slightly cloudy, cleared with lee. acetic acid and boiled. The iron precipitate was washed with boiling ammonium chloride water, dissolved in hydrochloric acid and re-precipitated. The trace of iron in the filtrates was thrown down with a drop of ammonia, filtered, washed and re-precipitated; the ammoniacal filtrates were then thoroughly acidified with acetic acid, and the copper, cobalt and nickel separated out by hydrogen sulphide. Finally the manganese was obtained as sulphide by precipitation with boiling ammonia and ammonium sulphide, and by heating the precipitate with sulphur in hydrogen by Rose's method. The manganese was thus found to be 46·22 per cent. The time necessary was two days. Volhard's method gives very excellent results, and several check determinations may be made with each solution; but it requires a long time. 1 gm. of the ferro-manganese was dissolved as above, but after taking up the residue with hydrochloric acid, sulphuric acid was added, and evaporation conducted until the fumes of the latter began to appear. Water was then added, and when all residue had dissolved the liquid was transferred to a litre flask and nearly neutralised with sodium carbonate. Zinc oxide was next added to throw down the iron, the flask filled to the graduation mark and well shaken. After the precipitate had subsided, the supernatant liquid was passed through a dry filter, and 200 cc. used for each experiment. Two drops of nitric acid were added to each test solution, which was then titrated with potassium permanganate (from the iron equivalent of which, that of manganese is found by multiplying by 0·2946). Results: 46·21, 46·33; mean=46·27%. Necessary time=10 hours; and this cannot well be lessened—but rather will be exceeded, unless the minima of acids are used for solution—since the evaporation and the subsidence of the MnO<sub>2</sub> precipitate formed during titration occupy so long a time. Hence this process will pro-

bably be used only as a check method. The modification tried at the West German Iron Works of precipitating with  $\text{ZnO}$  direct from hydrochloric solution, and then titrating without filtering is found not to give constant results. In Hampe's potassium chlorate process 0.3grms. of the alloy is dissolved in a 400cc. covered beaker with 25cc. of 1.18 sp. gr. nitric acid; 5grms. of chlorate are then added by degrees, and the solution is gently raised nearly to the boiling point during two hours. After dilution with hot water, the  $\text{MnO}_2$  precipitate is collected on an asbestos filter, thoroughly washed with hot water, and re-transferred with the asbestos to the beaker, where it is dissolved in 50cc. (accurately measured) of an acid ferrous sulphate solution (50grms.  $\text{FeSO}_4$  in 250grms.  $\text{H}_2\text{SO}_4$  and 750 water) and titrated with permanganate. 50cc. of the ferrous sulphate solution are at the same time titrated *per se* in another beaker; the difference between the two readings gives the amount of iron peroxidised by the  $\text{MnO}_2$ . The manganese equivalent of the permanganate is found by multiplying its iron equivalent by 0.491. Results: 46.49, 46.64, 45.92; mean = 46.35%; time = 10 hours. Five to six hours were taken up in washing the precipitate, to which the chlorate clings very tenaciously; again, the manganese is not always completely precipitated. Hence, of the two methods, Volhard's is distinctly the better. Pattinson dissolves 0.3grms. in aqua-regia and evaporates, redissolves in a minimum of cold water, just neutralises with dry calcium carbonate, and adds 50cc. chloride of lime solution (15grms. of chloride in one litre of water and filtered) and about 300cc. of boiling water, and heats to  $80^\circ$ . Now calcium carbonate is again added with stirring, until a slight excess is visible at the bottom of the beaker (when raised). A little of the clear liquor is to be tested for manganese with ammonium chloride and sulphide. If the solution should become red from manganic acid, the latter may be reduced by digestion with alcohol. The precipitate is then thrown on a filter, washed, and, with the filter, re-transferred to the beaker, dissolved and titrated as in Hampe's method. Results: 46.28 in each of two tests; lime =  $\frac{5}{4}$  hours (*i.e.*, accuracy is equal to Volhard's, but time only one-half). It has been said that copper, cobalt, or nickel would, if present, come down with the manganese as higher oxides; but the error due to this cause is very slight. If the copper precipitates as  $\text{CuO}$  no danger is incurred; if—and this is improbable—as  $\text{Cu}_2\text{O}$ , 1 equivalent of copper would only correspond to 0.44 equivalents of manganese; or to take an extreme case, 0.4% of copper in the alloy would cause an error in the manganese of only 0.17%. So, also, with nickel and cobalt, the amount reckoned as manganese is far less than the percentages of the foreign substances. Too high manganese results are to be traced to insufficient washing away of the oxidising chloride of lime. The washings should always be tested with sulphuric acid, potassium iodide and starch; it should not require more than two hours. The author has found no difficulty from the supposed influence of the yellow peroxide of iron solution in masking the red coloration indicative of the end of the reaction; nor does he find that the filter paper, by deoxidation of  $\text{KMnO}_4$ , interferes with the reading if it be taken immediately upon the first tinge of pink throughout the solution. If the solution should have a dark colour before titration, it may be decolorised by gently warming, or by a few drops of sulphuric acid. If the alloy contain a much larger percentage of manganese than of iron, pure ferric chloride should be added to make up the difference. Finally, the author makes a few additional remarks on the colorimetric process (see *Journ. Soc. Chem. Ind.*, 1883, 249), which is now in constant use in several foundries for small percentages of manganese (under 1%). The objection that some manganese may sometimes remain as peroxide with the binoxide of lead need not be feared, because with the same solution several fractions may be taken and nine or ten check experiments made, and the mean of these will be sufficiently correct. The author always makes at least three trials. And the process is very rapid, for one asbestos filter may be used in many experiments, so that while one is

filtering another is under treatment with lead peroxide. Certain special precautions should be observed in conducting this process: avoid using too great an excess or too little nitric acid; add  $\text{PbO}_2$  to the solution nearly boiling, and gently boil for a few minutes (always the richer the alloy in carbon the longer the time of boiling); thoroughly ignite the asbestos; let the asbestos plug be not too loose. This last hint is very necessary to be observed, for finely divided lead peroxide passing into the solution gives it a darker colour than it should have; if any should pass the filter, the solution should be re-filtered through fresh asbestos. But it is best to wash the peroxide by decantation, and not to throw it upon the filter; two washings with 1 or 2cc. of water will generally suffice.—W. G. M.

*On the Recovery of Tin and Iron from Tin-plate Scrap.*  
Dr. Czimatis. Chem. Zeit. 1884, 678.

It is not possible to obtain a complete separation of the iron and tin by proceeding according to the various methods which depend upon bringing the scrap to the melting point of tin, which is then removed in the molten state by mechanical means, such as revolving the scrap in a drum with sand, or by use of a centrifugating machine. The iron which is left contains so much tin as to be of little value for working up in the furnace. The salts of tin obtained by treating the scrap with acid solvents are also too much contaminated with iron to render an acid extraction process advisable. Better results, however, should be obtained with Reinecker's process, which depends upon the power which caustic alkali has of dissolving tin when an oxidising agent is present. According to this process, the scrap is cut up into small fragments, treated in revolving drums with caustic soda for the removal of grease, and thence transferred to other drums, to be acted upon by a solution of oxide of lead in caustic soda. The reaction which occurs may be expressed by the equation:  $\text{Sn} + 2\text{NaOH} + 2\text{PbO} = \text{Na}_2\text{SnO}_3 + 2\text{Pb} + \text{H}_2\text{O}$ , and since, to a great extent, this is prevented from taking place by atmospheric carbonic acid, Reinecker has devised (*Chem. Zeit.* 1884, 509) a suitable apparatus for the purpose. The lye containing stannate of soda is separated from the finely divided lead, the former to be worked up in the most convenient manner, the latter to be oxidised and employed in subsequent operations.—W. D. B.

*Cadmium in the Zinc Dust of Cillier.* Oesterr.  
Ztschr. 32, 365.

In the zinc works of Cillier, the amount of cadmium in the sieved dust has been found to be .302—356 p.c. and in the coarser residue .262 p.c. From an examination of the zinc in course of distillation the author concludes that the cadmium distills over in the first stages of the process, the greater portion of which has collected after 6 hours in the cooler parts of the receivers. The experiments will be continued with the view of obtaining the cadmium in sufficient quantity to be workable by the method adopted in the zinc works of Upper Silesia.—J. C.

*Determination of Manganese and Phosphorus in Iron and Steel.* M. Troilius, Chem. Centr. Bl. 1884, 717.

*Determination of the Manganese.*—The following quantities are used. For iron or steel 5grms. filings, for spiegeleisen 0.5grms., and for ferromanganese 0.2grms. The iron or steel is dissolved in dilute hydrochloric acid, and the solution evaporated just to dryness. Strong nitric acid of 1.42 sp. gr. is now added, and the whole boiled with gradual addition of potassium chlorate. The precipitate is then collected on an asbestos filter, employing snetion, washed with strong nitric acid and finally with water. In the case of spiegeleisen and ferromanganese the treatment with strong nitric acid and potassium chlorate should be repeated once or twice. The contents of the filter tube are transferred to a beaker and



treated with 100cc. of a solution of ferrous sulphate (titrated with potassium dichromate and potassium ferricyanide). The quantity of unoxidised iron is then determined with standard dichromate. The difference gives the quantity of iron oxidised by  $\text{MnO}_2$ . On multiplying this by 0.491, the quantity of manganese is obtained, thus:  $2\text{FeO} + \text{MnO}_2 = \text{Fe}_2\text{O}_3 + \text{MnO}$  or  $55 : 112 = 0.491$ . *Determination of the Phosphoric Acid.*—5grms. of filings are treated with strong nitric acid of 1.42 sp.gr., evaporated, and hydrochloric acid of 1.19—1.2 sp.gr. added towards the end of the evaporation. The mass is then boiled with hydrochloric acid, filtered, washed with dilute nitric acid, and finally with hot water. The filtrate is treated with 20cc. strong nitric acid, and 80cc. molybdate solution, obtained by dissolving 1 part molybdic acid in 4 parts ammonia of 0.96 sp.gr., and adding 15 parts nitric acid of 1.2 sp.gr. 20cc. of ammonia of 0.88 sp.gr. are now added. The yellow precipitate, after standing for 24 hours at  $40^\circ$  is filtered, washed with water containing molybdate solution and redissolved in ammonia. The solution is then precipitated with magnesia mixture, obtained by dissolving 11 parts crystallised magnesium chloride, and 28 parts ammonium chloride in 130 parts water, and adding 70 parts ammonia of 0.96 sp.gr. The magnesia precipitate is allowed to stand for 12 hours, filtered, and washed with a minimum quantity of a cold mixture of 1 part ammonia sp.gr. 0.96, and 3 parts water. It is then dried, ignited, and weighed as magnesium pyrophosphate (containing 28 p.c. phosphorus.)—D. B.

*Extracting Metals from their Ores, &c.* T. R. Jordan, London, J. N. Longden, Charters Towers, Queensland. Eng. Pat. 5236, Nov. 5, 1883.

THE inventors propose to treat gold and silver ores by breaking in a stone breaker, then pulverising in a machine capable of reducing the broken ore to an extremely fine condition; finally the powder is delivered by an air current into a receiver. In this chamber it may be separated by the air current into different degrees of fineness, specific gravity and value, and if necessary is concentrated therein, each grade being then passed by screw conveyors or other suitable means to an amalgamator. After passing through the amalgamators the tailings are subjected to a further process of separation by a blast of air which conveys them through a concentrating chamber, over which they are distributed, the particles of different density falling into the various divisions of the said concentrator and the waste being blown away.—J. T.

*Extracting Cobalt, Nickel, and Manganese from their Ores.* J. B. Readman, Glasgow. Eng. Pat. 5359, Nov. 13, 1883.

THE invention consists principally in treating the ores in a pulverised condition with one or more of the chlorides of iron, calcium, strontium, or magnesium, or with any other chloride which is soluble and otherwise convenient. The operation is effected either by fuming the ore and chloride, or by making them up into an emulsion with water and thoroughly agitating the mixture for some time, with or without the application of heat. To cobalt ore, finely powdered, a solution of, by preference, iron chloride is added, one part ore to one and a quarter parts of (anhydrous) chloride. The mixture is well stirred and evaporated to dryness, and the dry mass heated to a temperature sufficient to decompose the iron chloride; the mass is then lixiviated; the resulting liquid contains the chlorides of cobalt, nickel, and manganese, with only a small proportion of iron.—J. T.

*Manufacture of Steel.* W. Beardmore, Glasgow, and J. M. Cherrie, Glasgow. Eng. Pat. 5367, Nov. 14, 1883.

THE inventors propose to cast steel in shallow ingot moulds, open-topped or covered, so that the enclosed gases have a much shorter column of liquid metal to pass through before escaping than in the case of the deep moulds hitherto used either for open hearth or Bessemer steel.—J. T.

*Production of Zinc.* A. P. Price, London. Eng. Pat. 5416, Nov. 16, 1883.

ZINCIFEROUS materials are, together with suitable fluxes and reducing agents, subjected to the action of heat in a Cnpola blast furnace, so constructed that the products of combustion, together with the volatilised zinc, may be conducted into suitably arranged condensers.—J. T.

*Obtaining Copper from Cupreous Solutions.* A. P. Price, London. Eng. Pat. 5407, Nov. 16, 1883.

TO the solution containing copper, zinc in a fine state of division, such as zinc fume, is added, and the solution is agitated by the injection of air or steam, or by mechanical means. The precipitated copper is separated by filtration or decantation. The spent liquid may be used for extracting soluble copper salts, and after one or more operations may be treated with lime or other base to precipitate the zinc; the zinc compound thus obtained may be employed for the production of the finely divided zinc.—J. T.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*Solvent Action of Acetic Acid on certain Fats and Oils.* E. Valenta. Dingl. Pol. Jour. 252 pp. 296–297.

MANY animal and vegetable fats are more or less soluble in acetic acid. This property seems to offer advantages over previous methods of testing single fats. Equal parts of oil and acid are mixed and submitted to various temperatures. (1) Completely soluble at ordinary temperatures ( $15^\circ$  to  $20^\circ$ ): olive-kernel and castor oils. (2) Completely or almost completely soluble at temperatures between  $23^\circ$  and the boiling point of acetic acid: palm  $23^\circ$ , bay  $27^\circ$ , mace  $27^\circ$ , coco nut  $40^\circ$ , palm kernel  $48^\circ$ , elipsey  $64^\circ$ , green olive  $85^\circ$ , cacao nut  $105^\circ$ , gingerly  $107^\circ$ , pumpkin-seed  $108^\circ$ , almond  $110^\circ$ , cotton-seed  $110$ , rühl  $110^\circ$  (Hungarian, used to adulterate rape), yellow olive oil  $111^\circ$ , earth-nut  $112^\circ$ , and apricot kernel oils  $114^\circ$ , tallow  $95^\circ$ , American bone fat  $90$ – $95^\circ$ , cod-liver oil  $101^\circ$ , and press tallow  $114^\circ$ . The numbers give the temperature at which the cooling solution begins to be turbid. (3) Incompletely soluble at the boiling point of acetic acid: rape seed, rape, and hedge-mustard oils. It may be mentioned that acetic acid at  $50^\circ$  to  $60^\circ$  offers a good means of detecting the adulteration of mineral oil by means of resin oil, the former being almost insoluble, whilst the latter is very easily soluble.—J. T.

## XIII.—TANNING, LEATHER, GLUE AND SIZE.

*Contributions to the Chemistry of Tannins.* Dingler's Polyt. Jour. 253, 8, p. 340.

O. NASSE (Berichte der Deutschen Chemischen Gesellschaft, 1884, p. 1166) states that gallotannic acid, both in aqueous and alcoholic solution, is coloured a fine purple red by iodine solution in presence of neutral or acid salts, which in themselves neither colour nor precipitate tannin. The colour is fleeting and becomes a dirty brown, and the more quickly so the warmer the solution. Gallic acid and pyrogallol show a similar reaction, but no other of the more familiar tri-hydroxyl benzol derivatives (phloroglucin, querciglucon, and ellagic acid were tested); nor is it produced by any of the mono- or di-hydroxylised derivatives. It is only applicable to the examination of vegetable extracts when the tint produced is not masked, either by a colour already formed, or produced in the extract by the reagents employed. The latter is frequently the case, as in the roots of certain rosaceae, in myrabolams, etc.; acorns give a beautiful pure blue colouration, probably due to quercetin.

Perret (Bulletin de la Société Chimique, 1884, vol. 41, p. 22) to estimate tannin, boils the sample twice with water for 15 minutes, evaporates the extract to 100cc., filters hot, cools to  $70^\circ$ , and adds, while stirring, a solution of 1 part of dry albumen in 4 parts of water, so long as a precipitate is formed. He then heats the mixture to

boiling, and runs in a 20 per cent. solution of aluminium sulphate till the precipitate settles well. After cooling, the precipitate is collected on a weighed filter, washed, and dried on a plaster of Paris plate in the drying oven. The weight, the filter, the albumen, and the aluminic sulphate is now deducted, and the remainder taken as tannin.

A. Guyard (Idem. p. 336) states that pure air has no action on tannin in dilute aqueous solution, the change to gallic acid being effected by atmospheric ferments, probably without the agency of oxygen. In alkaline solution tannin is attacked by atmospheric oxygen directly. Gallic and tannic acids may be separated by lead acetate with excess of acetic acid, since the gallate, but not the tannate, is soluble in this solution. The lead tannate may afterwards be decomposed by sulphuric acid, or titrated with permanganate.

According to F. Musset (Pharmaceutische Centralhalle, 1884, p. 179) two tannins are present in oak bark, both of which are precipitated by gelatin, and are oxidised by permanganate, but in different proportions, so that the analysis is rendered inexact. Musset states that the oak tannin may be separated from the infusion by repeated agitation with acetic ether, while the oak-red tannin, the source of the oak-red, remains in the water solution. Since this process is tedious, Musset recommends titration with iodine. 0.1 to 0.07 gm. of the purest tannin is weighed into a bottle, holding 50 to 60cc.; dissolved in 20cc. of water free from air, and 20cc. of decinormal solution of iodine is added, and mixed, and the bottle filled with air-free water, and carefully closed. After standing over night, the contents are washed into a beaker and titrated with a solution of hyposulphite of soda and starch. The point of decolouration must be passed by about half a cc., and the mixture at once titrated back with free iodine, since this is unavoidable with bark extracts. The total iodine solution consumed, after deducting that corresponding to the hyposulphite, is that absorbed by the tannin. For the estimation of oak barks, an average sample of 15gm. is dried at 100°, and of this, 10gm. are weighed into a litre flask, which is filled with air-free water, and heated in the water-bath till the air is expelled from the bark. The flask is then closed, and the whole digested for many hours. It is then cooled, made up to the mark, shaken and filtered. In a 150cc. flask 100cc. of the extract is digested with 20cc. of iodine solution, and, the following day, is titrated in the same way as the tannin. This gives the total iodine consumption by 100cc. of extract. To a second quantity of 100cc. 20cc. of iodine solution is similarly added, the flask filled up with water, and allowed to stand all night. The iodised oak-red tannin which is precipitated is collected on a little vacuum filter under a layer of petroleum ether, washed with 20cc. of water free from air, and saturated with iodine, and dried in a stream of carbonic dioxide at 110°; 0.005 gm. is added for a trace dissolved in the wash water. The precipitate contains 7.8 per cent. of iodine. Further, 5gm. of zinc oxide is rubbed up with a portion of the bark extract, washed into a 300cc. flask, and filled to the mark with the extract, and carefully stopped. This is allowed to digest 24 hours, with frequent shaking, and at the end of this time is tested with gelatin or acetate of iron for tannin. The latter should give scarcely a perceptible change of colour. This filtrate now contains neither oak tannin nor oak-red tannin, and 100cc. is treated with iodine as before to determine that absorbed by the non-tannin constituents of the bark. By deducting this from the total iodine, that taken up by the actual tannins can be calculated, and the tannins reckoned on in terms of gallo-tannic acid. The quantity of iodine consumed by the oak-red tannin may be calculated from the precipitate with iodine, which contains 7.8 per cent. by adding an equal quantity, which is at the same time converted into hydrobromic acid. This doubled quantity deducted from the total consumption of both tannins gives that consumed by the oak tannin alone. The weight of the oak-red tannin may be determined by deducting the iodine contained in the precipitate of the iodised acid, and adding an equivalent quantity of hydrogen. German oak barks contain, according to Musset, 7 to 8 per cent. of oak tannin, and 6 to 10 per cent. of oak-

red tannin. Since the oak-red tannin also tans, the determination of the oak tannin alone, as Musset considers has hitherto been the case, is insufficient or practical purposes, and the more so, as the two tannins are probably of different practical values. Neubauer's difficultly soluble tannin is only a portion of the oak-red tannin which is more obstinately retained by the fibre; and the varied composition of the copper precipitates from oak bark is due to the varying proportion of the two tannins.

C. Böttlinger (Berichte der Deutschen Chemischen Gesellschaft, 1884, pp. 1041 and 1123) has investigated the tannin of the hemlock pine, which is used on an enormous scale for tanning in the United States, and is exported in the form of extract. A sample of this, which contained about 20 per cent. of tannin, was a thick brown and scarcely fluid mass, which dissolved in water to a clear brown liquid, leaving a red-brown amorphous residue. Infusions of hemlock tannin are of a darker colour than those of oak bark of equal strength. Hemlock tannin is with difficulty withdrawn from aqueous solution by agitation with acetic ether. It behaves with alkalis and sulphuric and hydrochloric acids like oak bark tannin, but the hemlock-red separated by the acids is decidedly more copper-coloured than oak-red. If the watery solution of the extract be shaken with bromine, it is temporarily darkened, and a copious yellow precipitate separates. This was filtered off, washed with water containing sulphurous acid, and dried in the exsiccator. The body may be further purified by crystallisation from alcohol, white flocks remaining undissolved. If the tannin be separated from the aqueous solution by agitation with acetic ether, and treated with bromine as above described, the bromine compound is at once obtained pure. From the analysis  $C_{20}H_{11}Br_4O_{10}$  was reckoned; therefore, the tannin must be  $C_{20}H_{11}O_{10}$ , and homologous with oak bark tannin,  $C_{19}H_{10}O_9$ . By warming the tetrabrom hemlock tannin with acetic anhydride the pentacetyl compound was obtained; suspended in chloroform with bromine, the body  $C_{20}H_{10}Br_6O_{10}$ . To prepare the bromine compounds of the bark tannins, the infusions must be made as far as possible in the cold, and allowed to stand so long as any fine powder separates. They are then gradually saturated with bromine, till the latter is in excess. The infusions, which at first become muddy and dark, suddenly clear and separate yellow powdery flocculent precipitates, which are filtered out and washed well with water, to which at first sulphurous acid is added. The bromine derivatives of hemlock, quebracho and mangrove tannins, and of leaf tannin are yellow red, those of oak and larch bark brownish yellow, and those of the chestnut, oak, and of terra japonica, quite light yellow. The bibrom-tannin of oak bark, the brom-hemlock, brom-quebracho, and brom-leaf tannins, lose at ordinary temperatures only traces, at most, of hydrobromic acid; other brom-tannins yield it when dry, at first rapidly, and then more slowly. The precipitates contain the following percentage of bromine:

Oak Bark .....	28.4	Mimosa .....	49.36
Hemlock Bark .....	43.6	Chestnut Oak .....	50.48
Quebracho Wood .....	41.5	Terra Japonica .....	53.2
Mangrove Bark .....	42.15	Larch Bark .....	52.8

They are soluble in alcohol and glacial acetic acid, but not in ether, in which, however, they deliquesce. They are changed by concentrated hydrochloric acid, at a temperature of 190°-100°, into coaly masses, which are anhydrides. The brom-tannins lose bromine as hydrobromic acid, as well as water, carbonic acid, and a gas burning with a green flame. The whole of the brom-derivatives react in alcoholic solution with hydroxylamine chloride, so as to produce nitrogenous substances, which, on boiling with hydrochloric acid, separate hydroxylamine. Acetic anhydride converts the brom-derivatives of these tannins into acetyl-compounds, the reaction with those richer in bromine taking place with extraordinary energy and with loss of hydrobromic acid. This is the case with the bromine derivatives of mimosa, chestnut-oak, terra-japonica, larch, and mangrove bark. The acetyl-compounds are bright yellow, and lighter in colour than the original substance. Concentrated cold hydrochloric acid does not combine with them. Phosphoric penta-chloride



becomes hot, and evolves hydrochloric acid when mixed with them, but the compounds obtained by after treatment are very similar to the original bodies. A clear solution of hemlock tannin is treated with concentrated hydrochloric or sulphuric acid, and then heated for a considerable time in the drying oven. First a brownish red mass separates and then a red powder; and round the upper part of the glass, which gets dry, a black ring forms, which must be removed. The precipitate must be filtered off, washed well with water, powdered, and washed again, dried, and then exhausted repeatedly with ether, and afterwards with hot alcohol, so long as this is strongly coloured by anhydro-compounds, and finally dried. The hemlock-red so purified is insoluble in ether and alcohol, but dissolves in caustic soda and warm sodic carbonate solutions. The larch brom-tannin purified by recrystallisation from alcohol,  $C_{21}H_{14}Br_5O_{10}$ , decomposes very easily with evolution of hydrobromic acid. It is readily soluble in dilute alkalis, alcohol, acetic ether, and glacial acetic acid, but only slightly so in hot acetic acid, although with acetic anhydride it combines with considerable heat, forming an acetyl compound and separating hydrobromic acid. In alcoholic solution it acts on chloride of hydroxylamine, yielding a nitrogenous derivative from which hydroxylamine is separated by boiling with concentrated hydrochloric acid. Concentrated hydrochloric acid at  $180^\circ$  to  $190^\circ$  converts the hexabrom-larch-tannin into a black mass free from bromine, but capable of being bromised; in the process hydrobromic acid, water, carbonic acid, and methyl are evolved. Bromine acts in chloroform only very slowly on the compound, producing a body soluble in ether. The acetyl derivative is a yellow powder, which dissolves slowly in dilute soda solution, readily in cold acetone and acetic ether, and with difficulty in hot alcohol. Its composition is  $C_{21}H_5Ac_6Br_5O_{10}$ . If infusion of larch bark be boiled with hydrochloric or sulphuric acid, anhydrides of the larch tannin corresponding to oak-red are produced, which are insoluble in water, and may be purified by treatment with ether and alcohol; the latter extracts no inconsiderable quantity of another body, which is also of anhydride character. Different products are obtained according to whether hydrochloric or sulphuric acids are used, the former yielding 28 per cent. of acetyl, and the latter 23.7, corresponding to  $C_{21}H_{13}Ac_6O_8$ . If the hydrochloric product be treated in chloroform with bromine, pentabrom-larch-red is obtained. The tannin which water extracts from the so-called terra-japonica (gambier) corresponds in composition and properties with larch tannin, differing only in the colour of some of its compounds; thus the bromised gambier tannin is quite bright yellow. The clear watery solution of the tannin yields, by heating with acids, reds, which are insoluble in alcohol, but which differ in colour and composition with the nature of the acid and the temperature employed. Tolerably concentrated hydrochloric acid at  $80^\circ$  yields a yellowish-brown substance which takes up four acetyl groups,  $C_{21}H_{14}(C_2H_5O)_4O_8$ . Concentrated, and at  $100^\circ$ , the product is brown, and takes up three acetyl groups only,  $C_{21}H_{14}(C_2H_5O)_3O_8$ . This terra-japonica red however is not quite pure, for if bromised in chloroform at  $20^\circ$  C. three substances are always obtained, of which only one is soluble in chloroform. This is produced in small quantity only; it is soluble in alcohol, ether, and glacial acetic acid, and crystallises in needles, which are not soluble in either water or sodic carbonate solution, but are changed by the latter to a bright blue-green body. This substance cannot be obtained from the bromised reds, which are insoluble in chloroform, even by a further treatment with bromine. The bromised red is separated by alcohol into a part rich in bromine and easily soluble in hydrate and carbonate of soda solution, and readily parting with bromine until it retains five atoms only; and a pentabrom body, which is readily soluble in dilute caustic soda, only sparingly in sodic carbonate in the cold. Böttinger considers oak bark tannin to be a methyl-ether of the condensation products of acetoacetaldehydes with tannin. The described substances contain the homologues of acetoacetaldehydes. The methyl is united to a carboxyl group.

According to a further communication of Böttinger's

(Berichte der Deutschen Chemischen Gesellschaft, 1884, pp. 1475 and 1503) digallic acid has the formula  $C_{14}H_{10}O_5 \cdot 2H_2O$ . Spite of the great similarity between digallic acid and tannin, they cannot at present be considered as identical, since the former produces no gallic acid on boiling with dilute hydrochloric acid; the two are probably isomeric only. Gallic acid containing water of crystallisation is insoluble in cold acetic anhydride, but if heated on the water bath solution slowly takes place. In two hours the gallic acid has disappeared, and in six the acetyl-compound is abundantly formed, and may be separated (by pouring the solution into much water) in long colourless prismatic needles, which melt at  $165^\circ$  to  $166^\circ$ , if slowly heated. They are scarcely soluble in water, but freely so in cold alcohol and acetic ether. They form a colourless solution in cold aqueous solution of sodic carbonate, while in caustic soda the solution is yellow-brown. Gallic acid deprived of water at a temperature of  $120^\circ$  gives also an acetyl-compound melting at  $150^\circ$ , which is similar to the pentacetyl tannin. Crystallised tannin, which is much more slowly attacked than gallic acid by fuming nitric acid, is completely soluble in acetic anhydride, and by warming the solution in the water-bath is converted into pentacetyl-tannin, which melts at  $137^\circ$ , dissolves easily in acetic ether, with difficulty in cold alcohol, and only very gradually in cold dilute sodic carbonate. The watery extracts of dividivi, algarrobbilla and valonia deposit much ellagic acid, recognisable by the red reaction with fuming nitric acid. If the watery extracts of algarrobbilla, dividivi and Hungarian galls (knoppem), are treated with concentrated hydrochloric acid, thick yellow precipitates are formed, which become pasty when warmed. Valonia extract forms flocks with hydrochloric acid only after half an hour's warming on the water bath. If these liquids are shaken with acetic ether, without attention to the precipitates contained in them, and the ether is then evaporated and the yellow residue acetylated, it is easy, by appropriate treatment of the acetyl-derivative, to obtain some colourless pentacetyl-tannin. This is especially abundant with sumach extract. The clear aqueous extracts of the above named materials, after settling and filtration, were evaporated in the water bath. In this process dividivi and algarrobbilla extracts froth strongly. As soon as the extracts harden on cooling they are removed from the water-bath and powdered. These extracts are brown to yellow, and give red liquids when treated with fuming nitric acid, but the colour reaction is not very distinct. The residues are insoluble in cold acetic anhydride, but on warming in the water-bath they gradually dissolve and acetylise. Hungarian galls leave a slight residue undissolved, which is filtered off. After eight hours heating the solutions are poured into cold water, and the dark coloured acetyl-compounds washed with water and dried. They are scarcely soluble in cold alcohol, more so in a mixture of acetic ether and alcohol, and most of all, though not completely, in acetic ether. If the acetic ether be evaporated a residue is left which contains from 43.2 to 44.1 per cent. of acetyl. Böttinger considers these substances as nearly allied, but not identical with acetyl-gallic acid and with acet-tannin. Both they and the portion insoluble in acetic ether dissolve readily in caustic soda solution, but not in dilute cold sodic carbonate solution. They are quietly soluble in fuming nitric acid. The residues of dividivi and algarrobbilla which are insoluble in acetic ether are white, and dissolve in soda solution with a violet colour.—H. R. P.

#### XIV.—AGRICULTURE, MANURES, Etc.

*The Preparation of Bi- and Tri-calcium Phosphate.* E. L. de Bouquet. Ger. Pat. 25151, June 5, 1883.

For this purpose the inventor dissolves the phosphates in hydrochloric acid of from  $10^\circ$  to  $12^\circ$  B. and collects the solutions, previously clarified by sediment, in a wooden vat provided with mechanical agitator. Solution of calcium sulphhydrate is then added to the contents of the vat until the white precipitate of bi-calcium phosphate begins to take a dark tinge from the co-precipitation of

sulphide of iron, experience having shown that sulphide of iron is not thrown down until practically the whole of the phosphoric acid has been separated. For the preparation of tri-basic phosphate the addition of calcium sulphhydrate solution is continued until the liquor in the vat has an alkaline reaction. The hydrogen sulphide liberated may be collected and made use of.—W. D. B.

### XV.—SUGAR, GUMS, STARCHES, ETC.

*Improvements in Sugar Making.* Dingl. Pol. Jour. 252, 287–293.

SUGAR extraction from molasses by Steffen's process is conducted as follows, according to the Brunswick Maschinenbauanstalt (Ger. Pat. 25376, Feb. 2nd, 1883). The molasses, syrup, &c., is diluted with cold water in a holder provided with an agitator; the temperature should not exceed 35°, nor the concentration 6 to 12 per cent. of sugar. To a measured quantity is added a definite quantity of lime powder, 50 to 100 parts per 100 of sugar, according to the nature of the materials. The whole is passed through filter presses to remove excess of lime. The filtrate is cooled as much as possible, by say a cold water worm, to be submitted to a second lime operation. The residue in the press, after being washed, is a waste product. The cooled solution above is again treated with lime powder,—100 parts of sugar requiring about 65 parts lime powder, the proper amount is readily ascertained; after rapid agitation the sugar falls out; the lye is passed through a second series of filter presses to separate the sugar-lime. The filtrate may be run to waste, or may be treated once again with lime powder. The sugar-lime in the presses is washed with cold water and placed in a wet mill, such as is used in existing works, and brought to a lye with beet-root sap, sugar solution, or water. In beet sugar manufactories this lye is used for extracting the raw sap; it can also be saturated and treated alone for sugar. Since this sugar-lime quickly yields calcium hydrate when mixed with dissolved free sugar, a part of the lime can be thus separated before adding an acid.

H. Briem gives a detailed account of sugar refinery practice.

E. Allary proposes to improve the illumination of the half-shadow saccharimeter by disarding the lamp, surrounding the instrument with an internally blackened paper tube whose end is closed with a translucent yellow paper, or glass. The observer surrounds the instrument with a black cloth, which should also envelop his head, and the tube is turned towards a bright wall.

K. Stuekenberg, Dessau (Ger. Pat. 29218, May 16th, 1883), has found that, in the production of strontium sugar from molasses by Scheibler's method, the excess of strontium hydrate required for the separation of the bistrontium saccharate acts only as free alkali. The saccharate is difficultly soluble in all alkaline liquids, consequently the excess of strontium hydrate can be replaced by potassium or sodium hydrate. To the sugar solution, for each mol. sugar, somewhat more than one mol. free alkaline hydrate is added, then a slight excess of strontium hydrate; on boiling the saccharate is thrown down. In the lye a little strontium remains and the added alkali remains, partly free and partly combined with organic acids. The strontium hydrate may also be added before the alkaline hydrate. On cooling the solution, strontium is almost completely precipitated as hydrate. The excess of strontium in the old process can also be precipitated by the addition of alkaline hydrate. The alkaline residues are converted into beet-root potash, and finally into alkali, to be again employed in the process.—J. T.

*On the Preservation of Beet-root.* Dingl. Polyt. Journ. 253 [9] page 391.

G. VIBRANS (Deutsche Zuckerindustrie 1884, 216) has observed that beet-root kept under soil to protect it from the frost contains 0.5 per cent. sugar less than if kept in a heap. As the temperature of the root under soil is from 4° to 5°C. higher in autumn than that in the heap, it seems preferable to preserve beet-root in heaps, well covered with moss, etc.—S. H.

*Melitose from Cotton Seed.* H. Ritthausen. Journ. Prakt. Chem. 29, 351–57. Königsberg.

BERTHELOT'S melitose,  $C_{12}H_{22}O_{11} \cdot 3H_2O$  was until lately only found in manna from Tasmania, where it exudes in drops from a species of eucalyptus. The author has found that the residues from the pressed cotton seeds contain a large quantity. By extracting with 80 per cent. alcohol a yield of 3 per cent. from these residues was obtained. The residues are heated with alcohol to 60–70°, colouring matters extracted with ether, and the remainder precipitated, after carefully diluting with water, by the addition of lead acetate. From the filtrate the excess of lead is precipitated with sulphuretted hydrogen and the filtered solution evaporated. After standing 10 to 12 days in the cold (0° to 3°) a crystalline mass separates and the mother liquors yield a further quantity. Melitose has a slightly sweet taste, easily soluble in water. The aqueous solution gives a stronger right polarisation than saccharine ( $\alpha_D = 117.4^\circ$ ). Alkaline copper solution and other reagents for glucose are not changed even on heating. By warming with dilute sulphuric acid a faint yellow colouration appears and the solution now reduces alkaline copper solutions, and the rotation of the inverted melitose is reduced ( $\alpha_D = 61.8^\circ$ , the elementary analysis agrees with that of Berthelot.

J. C.

*Citric Acid in Leguminous Seeds.* H. Ritthausen. Journ. Prakt. Chem. 29, 357–59. Königsberg.

The author discovered this acid in the seeds of *vicia sativa* (vetch) v. *faba* (bean) and different kinds of peas and white garden beans. The powdered seeds are digested with water and a little hydrochloric acid at the ordinary temperature for half an hour, and, after neutralising, evaporated and separated from insoluble substances by filtration. The acid is then precipitated with lead acetate and the precipitate decomposed with sulphuretted hydrogen, the excess of which is driven off. It is then heated with milk of lime until alkaline, filtered again and boiled. The precipitate is dried and decomposed with its equivalent of sulphuric acid. The citric acid crystallises from the solution.—J. C.

*Estimation of Water in Potato Starch.*—Chem. Zeit. 52, 934. From Ztschr. f. Spiritus ind. 7, 550.

SAARE has worked out a new method which, even in the hands of ordinary workmen, combines rapidity of execution with sufficient accuracy for commercial purposes. 100 grms. of the starch are rinsed into a tared measure-flask, which is then filled up to 250cc. with water, at 17°5C., and weighed. The weight of the empty flask is now to be deducted from this amount, and the percentage of water, corresponding to the difference, found from the following table:—

Weight found.	Water per cent.	Weight found.	Water per cent.
289.40	0	277.20	31
289.00	1	276.80	32
288.60	2	276.40	33
288.20	3	276.00	34
287.80	4	275.60	35
287.40	5	275.20	36
287.05	6	274.80	37
286.65	7	274.40	38
286.25	8	274.05	39
285.85	9	273.65	40
285.45	10	273.25	41
285.05	11	272.85	42
284.65	12	272.45	43
284.25	13	272.05	44
283.90	14	271.70	45
283.50	15	271.30	46
283.10	16	270.90	47
282.70	17	270.50	48
282.30	18	270.10	49
281.90	19	269.70	50
281.50	20	269.30	51
281.10	21	268.90	52
280.75	22	268.50	53
280.35	23	268.10	54
279.95	24	267.75	55
279.55	25	267.35	56
279.15	26	266.95	57
278.75	27	266.55	58
278.35	28	266.15	59
277.90	29	265.75	60
277.60	30		



Thirty tests gave, for the sp. gr. of perfectly dry potato starch, 1.650 as a mean, the extremes being 1.647 and 1.653, and the table is based upon these figures. The results are correct to 0.25 per cent., or to 0.5 per cent. if the normal temperature for the water be disregarded. Green starch, and other poorer qualities, yield also sufficiently accurate results by this method.—W. G. M.

*Improvements in the Manufacture of Farinaceous Food.* Julius Schweitzer, of 53, Loughborough Park, Brixton. Eng. Pat. 1884, April 22, 1884.

It has hitherto been found advantageous to introduce steam at the atmospheric pressure into the ovens employed for baking flour to be used in the preparation of farinaceous food. According to the present invention, it is proposed to conduct such baking operation in the presence of steam under a pressure of about three atmospheres, and it is claimed that by such a proceeding the starch granules are broken up more completely than when steam at the ordinary atmospheric pressure is employed. The apparatus consists of a strong iron chest, capable of resisting an internal pressure of three atmospheres, about 8ft. broad, 6ft. deep, and 12in. high, and provided top and bottom with steam jackets or shelves. When the oven is hot, and the pressure of steam in the steam jackets is from 10lb. to 20lb. per square inch, steam is allowed to enter the upper part of the oven, so as to fill the space between the flour and the top jacket with an atmosphere of heated steam, the pressure of which is gradually increased to from 35lb. to 45lb. per square inch. During the baking process, the steam is shut off about every half hour, and the flour well turned over to ensure its thorough contact with the steam. Precautions are taken to obviate the introduction of any condensed water, and the baked flour does not therefore become wet or pasty, but simply clammy. If desired, the action of the steam may be assisted by mixing the flour with a small known percentage of any suitable acid, such as phosphoric, hydrochloric, citric, or tartaric, and subsequently neutralising such acid by the addition of an alkali. The patentee lays no claim to the introduction of steam into the oven, nor to the apparatus employed, but to the invention of baking flour for the manufacture of farinaceous food in a close oven, and in the presence of steam under pressure.—W. D. B.

## XVI.—BREWING, WINES, SPIRITS, Etc.

*Thickening of Pasteurised Beer.* Chem. Zeit. 52, 937. From Amer. Chem. Rev. 4, 26.

ACCORDING to E. Siebel, beers which became turbid after pasteurisation showed greater acidity than unpasteurised beer; and since the precipitate of albumen with tannic acid is less soluble in acid solutions, this increased acidity might be regarded as the cause of the turbidity. The presence of a little silica in the precipitate led to the belief that it was derived from the flask; and it was found that hot distilled water attacked far more readily those flasks in which pasteurised beer had become turbid, than those in which it remained clear. Hence it may be assumed that by pasteurising in flasks of non-refractory materials silica is taken up, and gives rise to turbidity after heating.—W. G. M.

*Nitrogenous Substances in Barley and Malt.* C. Lintner (Zeitschr. f. Spiritus ind. 1883, 997), Pol. Journ. 251, 28.

THE author has investigated 15 samples of barley and the malt from them, in order to ascertain whether the amount of nitrogenous matter which they contain is any guide to the working of diastase on the malt. The process of malting is made with 200gr. of carefully washed barley, which is left to germinate about seven days, until the radicle attains a length of three-quarters that of the grain, and is then dried for six hours at 40°. 25gr. of the finely ground malt is digested in one litre of distilled water for six hours at the ordinary temperature.

One portion of the extract is used for the determination of the strength of the diastase according to the method of Kjeldahl, another for the determination of the amount of dissolved protein, by boiling and then adding Stutzer's reagent ( $\text{H}_2\text{CuO}_2$ ). On cooling, the mass is filtered and washed, and the nitrogen in the dried precipitate estimated by combustion with soda lime. The method of Kjeldahl consists in determining the amount of starch, which a given quantity of diastase will convert into maltose. The determinations were made with 5cc. of malt extract solution and 100cc. of standard starch solution for exactly 20 minutes at a temperature of 50° to 60°. At the end of this period the diastase is destroyed by boiling, and the amount of sugar determined by Fehling's solution. From the figures which the author obtained, it appears that the amount of nitrogen in the barley and malt is no criterion of the strength of the diastase; on the other hand, the quantity of soluble albumen varies in direct proportion to it. The determination of the amount of soluble albumen offers therefore a means of estimating the strength of the diastase. The above experiments are only for malt dried at 40°. Whether the same result holds good for kiln-dried malt requires further investigation.—J. C.

*Manganese in Wine.* Maumené. Rep. d. analyt. Ch. No. 13, 201, 1884.

THE author confirms the observations of Ostermeier, and detected manganese in 34 different wines. The amount varied so considerably even in one kind of wine that a fixed percentage could not be estimated. It appears therefore that manganese, like other mineral constituents of plants, though not exceeding a certain quantity for the same plant, varies largely within that limit, and is dependent on the quantity of mineral matter in the soil. J. C.

*Maize Malt.* A. Bänndl (Dingl. Pol. Jour. 252, p. 303)

REPORTS on Schuster's procedure for mashing with maize malt.—The starch converted was always less than in the case of barley malt, amounting to only 6 to 7 per cent. of that in the malt. The fermentation was unfavourable, which can only be attributed to a higher acid content (0.4–0.5 per cent.) which prejudiced the after action of the diastase. This excess of acid must be attributed to the higher temperature of the malting floor. The following operations are also favourable to the development of fungus, whose presence is shown by the microscope, consequently the yeast mash gave negative results. The process can only give good results under special conditions.—J. T.

*Raising Beer by the Generation of Carbonic Acid Gas.* A. J. Boulé. (A Communication from Charles Lauer, of Perpignan, France.) Eng. Pat. 5044, October 23, 1883.

THE object of this invention is to provide an apparatus whereby the carbonic acid gas is generated only when required, the evolution of gas being interrupted upon closing the drawing-off tap. The apparatus consists of two concentric cylindrical vessels, the outer of cast iron, the inner of porcelain. The outer cylinder is closed at the bottom, and serves to contain dilute sulphuric acid; the inner is open at the bottom, but joined gas-tight at the top, with a cylinder of tinned copper closed by a lid held down by a bridge fixed by bolts. In the lid is a stuffing box, holding a screw-threaded rod, which carries a cone-shaped vessel at its lower end. The conical vessel, which is intended to contain bicarbonate of soda, is perforated, and may be raised or lowered. A leading tube from the copper cylinder conveys the carbonic acid gas to a scrubber, whence it proceeds to a spherical receiver of bronze, and thence to the cask or barrel. When the apparatus is closed, the gas generated will force the sulphuric acid into the outer cylinder, and thus out of contact with the bicarbonate of soda.—W. D. B.

*Clarification of Beer.* Henry Harris Lake. (Communicated by Dr. Hugo Kunheim, of Berlin, and Dr. W. Raydt, of Hanover.) Eng. Pat. 5126, Oct. 29, 1883.

ACCORDING to the present invention, carbonic acid gas, produced by the expansion of liquid carbonic acid, is caused to act upon the surface of the beer contained in the vats, with the result that under the pressure of this carbonic acid the yeasty matters contained in the beer are precipitated. It may be necessary, according to the size of the vat and nature of the beer, to continue the pressure of carbonic acid gas for several days, repeatedly drawing and testing small samples to ascertain when the clarification is complete. When clarified, the beer, still under pressure of carbonic acid, is withdrawn from the vats into casks or barrels, and as there is practically no loss of carbonic acid, the beer remains free from yeasty matters, does not become turbid, and at the same time is so rich in carbonic acid that an after fermentation is rendered unnecessary for its preservation. The above process may advantageously replace the "Pasteur" method of fermentation.—W. D. B.

*Improvements in the Manufacture of Unfermented Drinks.* Edward Perrins, Warwick. Eng. Pat. 6707, April 23, 1884.

THIS invention has for its object improvements in the manufacture of unfermented and unintoxicating drinks from malt and hops, which shall remain good and without apparent deterioration for a considerable length of time. To make twenty-eight gallons of such beer, to which the inventor has given the name of "Salus," one bushel of malt is mashed twice over as in the ordinary course of brewing, twenty pounds of sugar added, and the mash run off into a copper and boiled together with three pounds of hops, for one hour and ten minutes. After boiling, it is put into a cooler, then run into a large open vessel; when cold, three ounces of salicylic acid are added, and the liquor is then fined. The fined liquor is charged with carbonic acid gas, as in the ordinary course for the preparation of aerated waters, and bottled. The above quantities of materials may be somewhat varied, but in all cases salicylic acid must be used in conjunction with carbonic acid gas.—W. D. B.

## XVIII.—SANITARY CHEMISTRY, DISINFECTANTS.

*Purifying Sewage and Foul Waters.* S. C. Dean, London. Eng. Pat. 93, January 1, 1884.

THE invention is said to consist of an improved method of aerating, oxygenating, filtering and purifying sewage and all foul waters, and apparatus for the same. The apparatus appears to consist of tanks, containing any suitable filtering material, the bottom of the tank being laid with perforated pipes, through which air, steam, or a gas is forced into the filter by any known means.

J. T.

*Filtering Sewage.* C. Waite, Reading. Eng. Pat. 7481, May 9, 1884.

THE inventor constructs sets of three tanks in descending levels. The sewage is passed into the first tank, where much of the matter in suspension is deposited. The liquid then passes by upward filtration into the second tank, where chemicals may be added if it be thought desirable. Similarly the fluid passes to the third tank, and from this through another upward filter it flows on to the surface of a downward filter, after which it runs away in a harmless condition. The deposit in the tanks is stiffened by the addition of fresh lime, and removed in carts or trolleys for utilisation.—J. T.

*Purifying Water.* W. Anderson, Westminster. Eng. Pat. 7665, May 13, 1884.

THE author causes the water to be purified to pass upwards through a vessel, preferably cylindrical with conical bottom. This vessel is charged with spongy

iron or metallic iron in a sufficiently divided state. The water enters the vessel from the bottom, and in an upward direction, with such velocity that it is able to keep the spongy iron or material in constant movement. The movement may be assisted by agitating blades fixed upon an axis, dipping down into the vessel and amongst the spongy iron or material which it contains. The purified water is drawn off from the top of the vessel, where the dimensions are such that the current is not sufficiently rapid to cause the spongy iron or material to be carried out of the purifying vessel. The escape of the spongy iron or material is further prevented by the use of a screen of perforated metal.—J. T.

## XIX.—PAPER, PASTEBOARD, Etc.

*Test for the Perviousness of Paper.* Aug. Leonhardi. Papier Ztg. 9625, Dresden.

THE author describes the following test for paper, by determining the permeating power of ink. One side of the paper is covered with a number of strokes of a solution of chloride of iron, containing 1.531 per cent. of iron, by means of a pen specially constructed for the purpose. After drying, a small quantity of ether of 0.726 sp. gr. and saturated with pure tannin is poured on the reverse side to that of the strokes, by holding the paper inclined. If the paper is impervious to ink no change will appear. In other cases, and according to the porosity of the paper, the reaction will become in a greater or less degree apparent. The pen nib is not movable, but so adjusted that both points, which must be rounded off, are at a distance of 1 mm. apart. The sides of the pen should be of ivory, horn, or ebonite. The chloride of iron solution gives a liquid, which, in penetrating power, corresponds to the average of the better sorts of writing and copying inks.—J. C.

*Waterproof Varnishes for Paper.* Papier Ztg., Pharm. C. H. 25, 107.

THESE varnishes are prepared as follows:—1. One part Damar resin; 4, 5, to 6 parts acetone are digested in a closed flask for two weeks and the clear solution poured off. To this 4 parts of collodion are added, and the whole allowed to clear by standing. 2. 30 parts white shellac are digested with 500 parts of ether, and to the solution 15 parts of lead carbonate are added, then shaken for some time and repeatedly filtered. 3. Five parts of glue are dissolved in 100 parts of warm water, and this solution spread on paper. After drying the paper is soaked for an hour in 10 per cent. solution of acetate of alumina and again dried, in order to give it a final glaze. 4. 120 parts of linseed oil are heated and poured into a mixture of 33 parts of quicklime and 22 parts of water, to which 55 parts of melted caoutchouc have been added, stirring all the time. The varnish is strained and used hot. 5. One part of guttapercha is carefully digested in 40 parts of benzene on the water bath, and the paper covered with it. This varnish can be drawn or written on.—J. C.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Quantitative Analysis of Morphine in Opium Powder and Extract.* Pharm. Post.; Schweiz. Wochenschr. f. Pharm. 22, 95-98.

IN the laboratory of G. Heli & Co. experiments have been made with the view of comparing the method of Bernhardt for estimating morphine with two others. 1. Method of Bernhardt: 1 grm. opium powder is rubbed with distilled water, the solution poured off and the operation repeated until a filtered portion is colourless and no meconic acid reaction with iron chloride noticeable. The filtered solution, amounting to about 40cc., is evaporated to 25cc. and shaken in a test glass with five drops of ammonia and 10cc. of ether. The layer of ether is then completely separated by decantation, and the remainder absorbed from the surface with bibulous



paper. The morphine, which precipitates as a crystalline powder, is collected on a weighed filter paper and the last traces of mother-liquor washed off with ether. The separation of the morphine was allowed to continue from four to five hours. Instead of washing the filter paper with ether, it was found preferable to use a small quantity of distilled water. 2. The second method, with few exceptions, was the same as described in the *Pharm. Germ.* ii.: 2grms. dried opium powder were extracted with distilled water, concentrated to 40cc., and treated with 5grms. of alcohol, 5grms. of ether, and 10 drops of ammonia. The mixture was left 12 hours after being well shaken. The morphine (separated) was collected on a weighed filter and washed with 2grms. of a mixture of alcohol, ether and water. 3. The third method is described in the *Osterr. Pharm.*: 2grms. of opium powder are left to macerate for 24 hours with 20grms. of a mixture of 2 parts dilute hydrochloric acid and 37 parts distilled water, then filtered and the residue washed with water. The filtrate is then heated with 8 parts of sodium chloride and allowed to stand 12 hours, then filtered, washed with NaCl solution and the filtrate treated with ammonia until it smells faintly. After 12 hours the precipitate is collected, dried and dissolved in dilute acetic acid and again precipitated with ammonia, washed with water and dried. By a comparison of analyses by the above three methods, the author gives the preference for accuracy and quickness to that of Bernhardt. A small correction is, however, necessary in the latter, as pure crystals of morphine are not always obtained. To obtain the correction, it would be necessary to estimate the percentage of impurities in a series of experiments.—J. C.

#### Quantitative Determination of Morphine in Opium.

Perger. *Jour. prakt. Chem.* 29, 97.

THE author compares the following methods of estimating morphine in opium: (1) Hager's method with Godeffroy's modifications. (2) The method of the *Pharmacopœia austriaca* Ed. VI. (3) Merk's method, and (4) a method which he recommends and describes in the following manner:—10 to 20grms. of finely divided opium are boiled with 15 to 30grms. caustic baryta and 150 to 200cc. water. The solution is filtered and the residue exhausted repeatedly with hot water until the filtrate ceases to produce the characteristic colour reaction with molybdate and sulphuric acid. The solution is then saturated with carbonic anhydride, and evaporated to dryness on a water bath. The residue is transferred to a flask and extracted with alcohol. The extract is evaporated, treated with 15cc. water, containing a small amount of ammonia, and allowed to stand for some time. The resulting precipitate is washed by decantation on a small tared filter. It is then dried at 40° in a small separating funnel. When dry the precipitate is digested on the filter with pure chloroform, re-dried and weighed. The weight obtained represents crude morphine, which may be purified by adding hydrochloric acid, morphine hydrochloride crystallising out, or by crystallising the crude product from alcohol. The following is a comparison of the percentages of morphine in opium obtained by the method referred to in the above. The impure products are marked thus\* :—

Opium.	E. Merk.	Pharm. austr.	Hager.	Perger.
I. ....	—	4.17*	1.630*	9.01
II. ....	5.99	2.04*	0.507*	8.37
III. ....	—	—	5.567*	9.10
IV. ....	9.32	0.253*	8.520	11.00
V. ....	1.72	0.300*	1.170*	3.68
VI. ....	13.57	2*	8.420	11.75

D. B.

#### On Colchicine. *Dingl. Polyt. Journ.* 253. [9] p. 391.

A. HOUDÉ (*Comptes rendus*, 1884, vol. 98, 1442) extracted for the preparation of crystallised colchicine, the seed of colchicum, with 96% alcohol. The latter is distilled from the filtered solution, the residue digested with a 5% solution of tartaric acid, and the acid solution shaken up with chloroform. After driving off the latter, crystals are left behind, which are purified by recrystallising out of a

mixture of chloroform, alcohol and benzene. The yield is 0.3% colchicine. It forms colourless prisms of a very bitter taste, fusing at 93° C. After drying at 100° C. they do not fuse until at 163° C. It is little soluble in water, glycerine and ether, very easily in alcohol, chloroform and benzene. Like colinine it appears to be a glucoside.—S. H.

#### Process for the Manufacture of Vanillin. *Dingl. Polyt. Journ.* 253 [9] p. 391.

HAARMANN and Reimer, Holzminden (Ger. Pat. 27992, 1883), have found that coniferin is converted by oxidation with chromic acid into glucovanillin. For this purpose a solution 10 parts coniferin in 200 parts water is treated at common temperatures with a solution of 8 parts chromic acid in as little water as possible. The mixture is left at rest for several days until a dirty brown precipitate has fallen out. A carbonate of an alkaline earth is then added, the liquor heated to a boil, and the last rest of chromium thus precipitated. The precipitate is filtered, and the filtrate concentrated to a small bulk, and treated with alcohol in excess. Small quantities of the sugar-vanillate of the alkaline earth are deposited along with glucovanillin. After distilling the alcohol off the filtrate, crystals of glucovanillin crystals are left behind, which fuse at 170° C. Coniferin dissolves in sulphuric acid with a deep violet, glucovanillin with a light yellow colour. The latter is split up into glucose and vanillin by emulsion in aqueous solution, or by boiling with dilute mineral acids,—sulphuric acid is best. From the solution the vanillin is separated by extracting with ether.—S. H.

#### Aseptol, a New Antiseptic. *Chem. Centr. Bl.*, 1884, 720.

THIS substance is an analogue of salicylic acid, its composition corresponding with the formula for hydroxyphenyl sulphurous acid,  $C_6H_4(OH)SO_2H$ . It represents a thick reddish fluid of 1.45 sp.gr., and has a faint odour resembling phenol. It does not possess the toxic action peculiar to phenol, and is therefore recommended for surgical and ophthalmic operations.—D. B.

#### On the Preparation of pure Sub-nitrate of Bismuth. M. Grossmann. *Chem. Zeit.* 1884, 677.

By combining the methods recommended by Hager and Deschamps, the author finds that it is possible to prepare sub-nitrate of bismuth, of snow-white appearance and absolutely free from arsenic, selenium, and other impurities. To 40 parts of officinal nitric acid, heated upon the water bath, 10 parts of commercial bismuth are added by degrees; tin and antimony remain undissolved as a brownish black powder; the solution is separated from this, boiled, allowed to digest for half an hour, and filtered through glass wool, oxide of iron being in this manner eliminated. The filtrate is poured into a solution of four parts of ammonium chloride in 200 parts of water, and the precipitate of oxide of bismuth contaminated only with co-precipitated zinc and lead is collected and well washed with water, whereby all the silver, arsenic, selenium, tellurium copper and nickel in solution are separated. For the removal of the co-precipitated zinc and lead, the moist precipitate is boiled with 15 parts of soda-lye in 15 parts of water, washed with warm water, and finally dried. Of the yellow oxide of bismuth thus obtained, 10 parts are dissolved in from 36.5 to 38 parts of nitric acid of 1.185 sp. gr., and the solution filtered through glass wool. One quarter of this solution is set on one side, and to the remaining three-fourths water is added until a turbidity becomes apparent. The quarter which was set apart is now mixed in, and the whole is poured slowly in a thin stream into 450 parts of boiling water. After a half hour the precipitate is filtered off, washed with 100 parts

of water, and dried at a temperature of 30°C. (*Note.*—The importance of bismuth sub-nitrate for medicinal purposes being absolutely free from tellurium, has recently been pointed out by Reisert. *Amer. Journ. of Pharm.* 56, 177. *Abstr.*).—W. D. B.

From this it may be seen that no such table can be very accurate, and this is the probable reason why such tables have not before been made. But for those who have much to do with ether, a table of even a fair degree of practical accuracy is far better than none at all; and

*Ether Table.* Dr. Squibb, *Pharm. Journ.*, 74-6, 3rd series.

TABLE OF SPECIFIC GRAVITIES of combinations of absolute ether and alcohol; the ether having a specific gravity of '71888, and the alcohol of '82012, both liquids at 15°C = 59°F., compared with water at 4°C = 39.2°F. as unity. Or, the ether of '71890, and the alcohol '82016, both liquids at 15.6°C = 60°F., compared with water at 15.6°C = 60°F. as unity.

This table is trustworthy to the third decimal place, and moderately accurate to the fourth.

The corrected columns are corrected for the expansion of glass only.

P.c. by Weight.	COMPARED WITH WATER AT 4°C. = 39.2°F. AS UNITY.							COMPARED WITH WATER AT 15.6°C. = 60°F. AS UNITY.		
	Weighed at—							Weighed at—		
	4°C. = 39.2°F.	15°C. = 59°F.		15.6°C. = 60°F.		25°C. = 77°F.		15.6°C. = 60°F.	25°C. = 77°F.	
	True.	Apparent.	Corrected.	Apparent.	Corrected.	Apparent	Corrected	True.	Apparent.	Corrected.
100	'73128	'71908	'71888	'71838	'71817	'70788	'70751	'71890	'70842	'70825
99	'73257	'72010	'72020	'71969	'71948	'70923	'70886	'72021	'70975	'70958
98	'73386	'72172	'72152	'72101	'72080	'71057	'71020	'72152	'71108	'71091
97	'73115	'72205	'72185	'72233	'72212	'71192	'71155	'72284	'71241	'71224
96	'73644	'72138	'72418	'72364	'72343	'71326	'71289	'72416	'71374	'71357
95	'73764	'72564	'72514	'72490	'72469	'71457	'71419	'72541	'71506	'71489
91	'73884	'72690	'72670	'72616	'72595	'71589	'71551	'72666	'71638	'71621
93	'73904	'72844	'72794	'72742	'72721	'71721	'71683	'72792	'71770	'71753
92	'71124	'72938	'72918	'72868	'72847	'71852	'71811	'72918	'71902	'71885
91	'74215	'73061	'73044	'72994	'72973	'71882	'71944	'73043	'72033	'72016
90	'74366	'73190	'73170	'73121	'73100	'72113	'72075	'73168	'72161	'72147
89	'74487	'73215	'73195	'73248	'73227	'72215	'72207	'73298	'72295	'72278
88	'74608	'73110	'73420	'73374	'73353	'72374	'72336	'73428	'72426	'72409
87	'71728	'73557	'73537	'73494	'73473	'72495	'72457	'73547	'72547	'72530
86	'74847	'73674	'73654	'73614	'73593	'72617	'72579	'73666	'72669	'72652
85	'71968	'73791	'73771	'73734	'73713	'72739	'72701	'73785	'72794	'72774
84	'75086	'73908	'73888	'73854	'73833	'72860	'72822	'73904	'72912	'72895
83	'75193	'74028	'74008	'73974	'73953	'72982	'72944	'74022	'73034	'73017
82	'75299	'71149	'71129	'74095	'74074	'73103	'73065	'74141	'73156	'73139
81	'75406	'71270	'74250	'74216	'74194	'73224	'73186	'74260	'73278	'73261
80	'75512	'74390	'74370	'74336	'74314	'73346	'73307	'74378	'73400	'73383
79	'75634	'71508	'74488	'74453	'74431	'73469	'73430	'74495	'73523	'73506
78	'75756	'74627	'74606	'74570	'74548	'73592	'73553	'74612	'73645	'73628
77	'75878	'74746	'74725	'74687	'74665	'73715	'73676	'74729	'73767	'73750
76	'76000	'74864	'74843	'74804	'74782	'73838	'73799	'74846	'73890	'73873
75	'76127	'74994	'74970	'74934	'74912	'73970	'73931	'74975	'74022	'74005
74	'76255	'75119	'75098	'75063	'75041	'74102	'74063	'75104	'74154	'74137
73	'76383	'75247	'75226	'75193	'75171	'74234	'74195	'75233	'74286	'74269
72	'76510	'75374	'75353	'75322	'75300	'74366	'74327	'75362	'74418	'74401
71	'76640	'75504	'75483	'75452	'75430	'74501	'74462	'75492	'74548	'74530
70	'76770	'75634	'75613	'75582	'75560	'74635	'74596	'75623	'74687	'74669

The absolute ether actually obtained by the writer is the basis of this table, and the specific gravities were taken by means of the flask. For diluting the ether an alcohol was prepared of a specific gravity of '82012 at 15°C., water at 4°C. being taken as unity, or '82016 at 15.6°C., compared with water at 15.6°C. as unity, such alcohol corresponding very closely to 90.75 per cent. absolute alcohol and 9.25 per cent. water.

since the number of those who handle and use ether is constantly and rapidly increasing, and will increase more rapidly still as it is more cheaply produced and its uses extend, such tables will become more and more useful.

The dilutions for the table were commenced with the absolute ether of this writer, and carefully adjusted alcohol of '82016, but from shaking together, and from



other exposure to moist air, both the ether and the alcohol of the mixtures became slowly weaker throughout the range of the table, and no attempt was made to prevent this, from considerations mentioned above. Dilutions were made and accurate specific gravities taken for each difference of 2 per cent. down to 88 per cent., and below that for each difference of 4 per cent., all the other specific gravities being supplied by interpolation. And as with alcohol so with ether, two standards of unity are in common use; specific gravities are given by both these standards, namely water at 4° C. and at 15° 6 C.

## XXI.—EXPLOSIVES, MATCHES, Etc.

*The Manufacture and Application of Explosives.* Dingl. Polyt. Journ. 253, 70, 1884.

THE explosive pancrastite, a mixture of liquid peroxide of nitrogen with certain hydrocarbons, or fatty substances, or the nitro-derivatives of the same, the invention of which is claimed by E. Turpin, and concerning which a considerable degree of interest has recently been displayed, can scarcely lay claim to novelty, or be considered likely to obtain widespread application. Some ten years ago, H. Sprengel described and patented in England an explosive of the same nature as pancrastite, from which it differed only in the fact that nitric acid was employed in place of liquid peroxide of nitrogen. The inconveniences which prevented Sprengel's explosive from being usefully applied are connected also, but to a still greater degree, with pancrastite. Some of the most serious drawbacks to the employment of the latter are the instability and disagreeable nature of the liquid peroxide of nitrogen, which boils at 22°C.; the impossibility of enveloping the cartridge in anything but glass; the trouble and danger to health attendant upon the mixture of the two ingredients, and the corrosive action of nitrogen peroxide upon the copper shell of the detonator. Moreover, the risks attendant upon the transport of such an explosive would be sufficient in themselves to prevent its distribution and employment.

An application of illumination by electricity has recently been made at the Hanm works of the Vereinigten Rheinisch-Westfälischen Pulverfabriken. The current is derived from a Gülicher dynamo of 130 ampères and 65 volts; two arc lights well removed from the danger buildings illuminate the exterior; the engine rooms are provided with incandescent lamps of 8 to 32 candle power, as are also the danger buildings, where the power of the lamps is from 16 to 18 candles.

An accidental explosion, which occurred in a slate quarry near the railway station of Cerbère, Prussia, points to the necessity for ascertaining with certainty whether a mine has been entirely exploded, or otherwise, by the first detonation. In this case a shaft had been sunk in hard slate of laminar structure to a depth of 8m. in a vertical direction; borings in a horizontal direction were now made right and left, and 660 kilos of dynamite filled into these horizontal chambers. The charge was detonated by the aid of a high-tension fuse, but its explosion performed but little work; the unfavourable result, however, was ascribed to the structure of the slate. Some three months afterwards, during the carrying on of blasting operations in the vicinity of this mine, a violent explosion took place, and fragments of stone were hurled on the railway 80m. distant, doing considerable damage to the rolling stock. It would thus appear that a portion of the charge in the first mine had escaped detonation, but that this was brought about three months afterwards by the vibrations caused by the explosion of other charges of dynamite in the vicinity. Accidents such as this may be avoided by detonating the charge in more places than one, and by the careful examination of the crater after the explosion has taken place.

Windsor, in conjunction with several others, has recently devised a so-called dynamite-cannon (*Scientific American*, 1884, p. 214), the merits of which have been investigated by a Commission, acting under instructions from the United States Government. This cannon consists of a strong steel tube, 1220m. long, of 5cm. internal diameter, and 6·5mm. thick, resting upon a light steel carriage

of such construction that the tube may readily be pointed in any desired horizontal or vertical direction. The projectile consists of a tapering wooden plug, the hindmost portion of which fits closely in the bore of the tube, and whose other extremity carries a thin metal shell filled with dynamite. In front of this metallic case is a mass of some soft substance, in which a detonator is embedded. A conical cap, with a rod attached to it, is fitted in front of all, and the rod, being in contact with the detonator, brings that to explosion as soon as the projectile meets a hard body. Motion is transmitted to the projectile by means of compressed air, the entry of which into the tube is regulated automatically, in such a manner that the pressure gradually increases until the projectile leaves the mouth of the cannon. In an actual experiment, it was found that, with an air pressure of 31 kilos per square cm., the distance traversed by a projectile fired from a 2-inch cannon was 2,000m. Since compressed air is the first requirement for this cannon, its employment is obviously confined to such positions where compressed air is readily obtainable. As to the reliability of the arrangements for the introduction of compressed air into the tube, and as to the certainty of explosion of the charge when the projectile strikes obliquely, further data are requisite.

From the eighth report of Her Majesty's Inspector under the Explosives Act, some idea may be gained of the condition of the English explosives industry in the year 1883. At the close of that year, there existed in Great Britain 104 factories for military and industrial explosives, and 13 for the preparation of fireworks. The number of magazines was 329, that is nine more than the number in 1882; the number of stores in April, 1883, licensed for quantities of powder, not exceeding two tons, or of dynamite and explosives of the same class not in excess of one ton, was 2,108, that is 63 more than in 1882. The number of expense magazines was 19,386, or 3,717 more than in 1880. As compared with the imports during 1882, there was also a marked increase in the quantities of dynamite and gunpowder imported during 1883. Of accidents there occurred 172 in all, resulting in the deaths of 39, and the wounding more or less seriously of 109 persons. Forty-eight of the accidents were simply due to the explosion of single cartridges, and were not attended by any serious results. The following table presents an analytical view of the circumstances under which the 172 accidents took place:—

Name of Explosive.	Accidents took place during				Total.
	Preparation.	Storage.	Use.	Transport.	
Gunpowder.....	26	1	26	1	54
Dynamite.....	2	—	22	—	24
Gun Cotton.....	6	—	9	—	15
Mercury Fulminate	1	—	—	—	1
Ammunition.....	66	—	4	—	70
Fireworks.....	5	—	1	—	6
Different Explosives	—	—	2	—	2

Of the 26 accidents which occurred during the manufacture of gunpowder, 19 were in the incorporating mills; only one man, however, being injured, and that by fire. A series of experiments has been conducted with a view of ascertaining whether the reserve store of cartridges in machine guns was liable to suffer detonation as a whole by the shock imparted by the discharge of one. It was found that the metallic cartridges, with a charge of 40·5grms. for the Nordenfält 1-inch machine guns, were not liable to be detonated *en masse*, but that in the case of the Nordenfält, Hotchkiss or Armstrong rapid delivery 6-pounder, the discharge of one cartridge was sufficient to bring about the explosion of the remainder, a fact readily understood when it is remembered that the charges for those three guns are respectively 1·247, 0·886, and 0·964kgms. Some experiments were also made as to the possibility of bringing dynamite to complete detonation by means of detonating mixtures, not containing any fulminate. The following mixtures in charges of 6·5 grains were tried—

- 1 potassium chlorate, 1 sulphide of antimony.
- 1 potassium chlorate, 1 sugar.
- 1 potassium chlorate, 1 sugar, 0·4 realgar.
- 4 potassium chlorate, 1 sugar, 1 realgar.
- 2 potassium chlorate, 1 lead ferrocyanide.
- An ordinary triple detonator, with a charge of 1·9 grains.

The dynamite was occasionally detonated by (a) and (e), and always so by (d) and (c), but (b) was found to be insufficient. With cellulose ("lignine") dynamite, all the above detonating compositions, (a) excepted, were found to be efficient. Experiments were made to ascertain whether old charcoal, after being wetted, was liable to spontaneous ignition on drying. This was found not to be the case. It has also been proved by direct experiment that gunpowder, brought in contact with iron at a temperature of 100°C., in either dry or moist air, is not liable to spontaneous inflammation.—W. D. B.

*Improvements in the Manufacture of Gunpowder and like Explosive Compounds.* Thorsten Nordenfelt, of Westminster, and Victor Algernon Meurling, of Christianstad, Sweden. Eng. Pat. 6514, April 18, 1884.

IN order to obviate the necessity for a prolonged mechanical incorporation of the constituents of gunpowder, and also to obtain of these a more thorough admixture than is possible by mechanical means, the carbon or carbonaceous matter is first thoroughly mixed with the proper amount of a concentrated solution of sulphur in carbon bisulphide, in a closed vessel provided with a mechanical agitator. When the mixture is complete the solvent is distilled off, and the recovered solvent employed for subsequent operations. The dry mixture of carbon and sulphur is then repeatedly saturated with an aqueous solution of potassium nitrate, the water being evaporated off at a gentle heat after each addition of the solution. When the proper amount of potassium nitrate has been introduced, the powder mixture is evaporated to dryness, and may then be finished according to the usual methods.—W. D. B.

## XXII.—GENERAL ANALYTICAL CHEMISTRY.

*Paratoluidine Sulphate as a Test for Nitric Acid.* A. Longi, Gaz. Chim. 13, 465.

THE red colour produced by nitric acid in a solution of paratoluidine sulphate and sulphuric acid is applicable to the detection of very small quantities of that acid.—A. S.

*A New Reaction of Benzdine.* By P. Julius. Monatsh. f. Chem. 1884, 193.

IN adding potassium bichromate solution to a concentrated solution of this base, a deep blue crystalline precipitate is immediately formed, which proved on investigation to be a chromic salt, having the composition represented by the formula  $C_{12}H_8(NH_2)_2CrO_4$ . The formation of this chromate affords a delicate reaction, occurring with very dilute solutions of the base. With a solution of 1 part in 50,000 the blue precipitate can be obtained on warming. The isomeric diphenylene gives a similar reaction, also in very dilute solution.—C. F. C.

*Detection of Nitric Acid in presence of other Acids which disguise its Reactions.* A. Longi, Gaz. Chim. 13, 468.

THE method is specially contrived for the detection of nitric acid in presence of hydrobromic, hydriodic, chloric, bromic, iodic, chromic and other acids. The solution which is to be tested for nitric acid, if acid, is neutralised with sodium carbonate and treated with excess of a solution of sulphurous acid. The solution is then gently heated to remove most of the sulphur dioxide and neutralised with sodium carbonate. In this way, acids rich in oxygen, such as chloric, bromic, iodic and chromic acids, are reduced. The liquid is heated to boiling, the insoluble metallic oxides filtered off, and the filtrate boiled with acetic acid and lead dioxide to expel bromine and iodine. The coloured liquid is filtered, and any lead which has gone into solution precipitated by sodium sulphate. The filtrate is evaporated to dryness on a water-bath, and in the residue nitric acid may be detected by the usual tests.—A. S.

*Estimation of Nitrous and Nitric Acids alone or when mixed.* A. Longi, Gaz. Chim. 13, 469.

THE method, which is a volumetric one, depends upon the known decomposition of nitrous acid by ammonia and amides into nitrogen and water, and the reduction of nitrous to nitric, by the action of arsenious acid. To determine nitrous acid the solution of the nitrite is decomposed by acetic acid and urea in a flask of 200cc. capacity, and the nitrogen collected and measured in the usual apparatus, which is connected with the flask. In the same apparatus nitrates are to be reduced by a hydrochloric acid solution of arsenious acid, and then decomposed by urea. The determination of the two acids can be combined in one operation. According to the author's examples, the process gives good results.—A. S.

*Qualitative Analysis with small quantities.* H. Hager, Pharm. Centralb. 23, 251-54.

THE usual complicated and wasteful method of testing pharmaceutical preparations can be just as well accomplished with a drop of the reagent, and the substance to be tested. The following apparatus and reagents are needed: (1) Eight or nine small bottles, holding 10-20cc., with glass stoppers drawn out into rods, so that they can be used for transferring a drop of the liquid to a test paper or glass; (2) six small bottles of 25cc. with caoutchouc stoppers, through which a glass rod is fixed. These are for solutions such as barium chloride, oxalic acid, sulphuric acid, etc.; (3) test papers prepared from good filter paper of litmus, turmeric, indigo carmine for nitric acid, and caustic alkalis, rosaniline paper for alcohol, prussiate of potash, paper for the different metallic salts and papers prepared with potassium sulphocyanide, sodium sulphite, potassium iodide, iodate, lead acetate, tannin, etc.; (4) two strips of brass for the detection of arsenic in the form of arsenic or arsenious acid. The hydrochloric acid solution is heated with oxalic acid solution or the ammoniacal extract saturated with hydrochloric acid and oxalic acid added. The latter effects the reduction of arsenic to arsenious acid. One drop of the solution is placed on the brass strip and evaporated to dryness at a gentle heat over the flame of a lamp. On washing with water, a permanganate coloured spot indicates the presence of arsenic. Nitric acid or nitrates, sulphuric acid or free ammonia should not be present in the solution to be tested; (5) three or four glass plates of clear, colourless and thin glass, whereon to make reactions or to evaporate liquids; (6) piece of platinum foil and wire. In testing, two drops of the liquid are placed on one of the glass plates and, close to them, a drop of the reagent. Then the two liquids are mixed, and any change in the transparency or colouration will be easily observed by placing the glass over white or black paper. The plates can be used also to distinguish volatile from non-volatile compounds by warming one or two drops of solution over a lamp to dryness. The smallest amount of residue can be at once detected under the microscope, which will also show whether it be crystalline or amorphous. The test papers are prepared by drawing them through a moderately concentrated solution of the reagent placed in a plate, to the depth of 1 or 2mm., and drying in a warm place.—J. C.

*New Tests for Nitric and Nitrous Acid Nitrates and Nitrites.* H. Hager, Pharm. Centralb., 23, 289-290.

IN place of the ferrous sulphate and sulphuric acid method the following may be adopted. The colourless solutions of the salts are strongly acidified with HCl, more especially where organic acids are present, then an amount about the size of a pea of powdered ferrous sulphate added to 3-4cc. of the acid solution. In presence of large quantities of nitrates or nitrites the liquid turns deep yellow. If it remain colourless, heat without stirring almost to boiling, and the presence of small traces of the nitrogen acid is indicated by an immediate yellow colouration. To test caustic alkalis an amount about the size of a bean is dissolved in 2cc. of water and treated with excess of HCl, and the ferrous sulphate



added as above. Nitrates or nitrites produce a yellow colouration. To test concentrated sulphuric acid, five drops of the acid are transferred to a piece of glass placed over white paper, and a pinch of the white ferrous sulphate dropped in the middle of the liquid. The same yellow colouration is produced. The colourless crystals of phenol are a delicate reagent for the nitrogen acids. If a phenol crystal be dropped into concentrated  $\text{H}_2\text{SO}_4$  it dissolves to a colourless solution. Should nitrates or nitrites be present, colours, varying in intensity from red to green, at once appear. Organic acids must be strongly acidified with hydrochloric acid, some crystals of phenol added, and the whole heated to  $80^\circ\text{--}90^\circ\text{C}$ . Similar deep colourations are produced. Both the above methods may be used with small quantities. To test ferric salts—for example, liq. ferrisulphurati—2 to 3cc. are placed in a test tube, then 1 to 2cc. of water and 2 to 3cc. pure hydrochloric acid, and, after mixing, 3 or 4 crystals of phenol. On warming to  $80^\circ$  to  $90^\circ\text{C}$ . the mass darkens and becomes turbid in presence of nitric acid. The presence of hydrochloric acid prevents the phenol reaction with ferric salts.—J. C.

## New Books.

**ANALYSIS OF MILK, CONDENSED MILK, AND INFANTS' MILK-FOODS, WITH SPECIAL REGARD TO HYGIENE AND SANITARY INSPECTION.** By DR. NICHOLAS GERBER, Manager of the American-Swiss Milk Product Company (Limited), New York, &c., &c. Translated from the Revised German Edition, and edited by Dr. HERMANN ENDEMANN, editor of the Journal of the American Chemical Society, &c., &c. Illustrated by 19 plates. New York, 1882. London: Trübner & Co.

THE editor claims in his preface that "this book is a laboratory guide, which enables even beginners, and all such who cannot make the subject of milk-analysis a speciality, to cope with this otherwise complicated task." The book is an 8vo. volume bound in cloth, containing 101 pages, table of contents at beginning, and 19 wood-cuts interspersed in the text. The subject-matter is subdivided as follows:—Normal cows' milk; physiological and other causes influencing the secretion; physical analysis of milk; microscopical examination; specific gravity; chemical analysis; abnormal cows' milk; milk of other animals; condensed and preserved milks; woman's milk; infants' milk-foods in powder; government control of the milk supply; determination of the fat; properties of normal cows' milk and market milk; the aim of government control of the quality of the milk supply; general rules for a rational milk inspection; proposition stating the main points for an ordinance regulating the sale of milk in large cities; propositions for regulations governing producers of milk and milk dealers; list of apparatus and chemicals required for the analysis of milk and infants' milk-foods.

**A COURSE OF QUALITATIVE CHEMICAL ANALYSIS.** By the late WILLIAM GEORGE VALENTIN, F.C.S. Revised and corrected by W. R. HODGKINSON, Ph.D. (Würzburg), and H. M. CHAPMAN. 6th Editor. London: J. and A. Churchill, New Burlington Street, 1884.

THIS is an 8vo. volume bound in cloth, in style and size resembling the former editions of this well-known work. It contains 305 pages of subject matter, map of spectra, and alphabetical index, and it is also illustrated by 17 wood-cuts. At the close of the several chapters there follow sets of "Questions and Exercises." The whole work is divided into eight chapters and five appendices, the 1st treating of

the rare metals, the 2nd of the various reagents, etc., 3rd, table shewing the solubility of salts in water and in acids, 4th, schemes for recording analytical results, and 5th, chart of spectra.

**A TREATISE ON THE PURIFICATION OF COAL GAS AND THE ADVANTAGES OF COOPER'S COAL-LIMING PROCESS.** By R. P. SPICE, M. Inst. C.E. London: E. and F. N. Spon, 16, Charing Cross. New York: 35, Murray Street.

THIS is an 8vo. volume, bound in cloth, and containing 79 pages of subject-matter, and in addition three appendices, and a tabulated statement of results referred to on page 2 of the text. The work is illustrated by 10 wood-cuts, and in its table of contents the subject-matter is subdivided as follows:—Modern history of the purification of coal gas; Cooper's coal-liming process; London gas; fallacies exposed; concluding remarks; appendices.

**THE ALKALI MAKERS' POCKET-BOOK.** Tables and Analytical Methods for Manufacturers of Sulphuric Acid, Nitric Acid, Soda, Potash, and Ammonia. By GEORGE LUNGE, Ph.D., Professor of Technical Chemistry, Zurich, and FERDINAND HURTER, Ph.D. London: George Bell & Sons, York Street, Covent Garden, 1884.

SMALL 8vo volume, bound in cloth, and containing preface, table of contents, preliminary notice, and 164 pages of subject-matter, including a table of errata, and an alphabetical index. The matter is arranged as follows:—*General Tables* and *Special Part*, which contains, concisely given, special analytical methods for valuing or otherwise determining the chief products of the following branches of industry. (1) Fuel and furnaces; (2) sulphuric acid manufacture; (3) salt-cake and hydrochloric acid; (4) bleaching powder and chlorate of potash manufacture; (5) soda ash manufacture; (6) sulphur recovery; (7) nitric acid manufacture; (8) potash manufacture; (9) ammonia manufacture. Appendix: (A) Preparation of standard solutions; (B) rules for sampling; (C) comparison of the hydrometer degrees, according to Baumé and Twaddell, with the specific gravities; (D) value of alkali per ton. The appearance of the German equivalent of this work, viz., "Das Taschenbuch für die Soda, Pottasche, and Ammoniak Fabrikation," has already received due notice (See this Journal II. [3] 141), but this, the English translation, besides being more generally accessible to British manufacturers, contains additions coming from the pen of Dr. Hurter, on the Deacon's process, and on chimney-testing. This gentleman has been at great pains, moreover, to convert all the tables containing metrical measurements and data, into their corresponding British equivalents.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1884.

12585 J. O'Neill Mackie, Liverpool. Improvements in sampling and testing apparatus for spirituous liquors or other liquids. September 19

12593 T. J. Barnard, Harlesden. Improvements in the economical concentration of ores, minerals, and metallic substances. September 19

12595 H. J. Haddan, Westminster. Improvements in the treatment of slag and in recovery of certain materials used therein. Communicated by G. Deumelandt, Potsdam. September 19

12617 J. Kirkaldy, Poplar, Middlesex. Improvements in surface condensers and heaters. September 19

- 12643 W. Donaldson, J. Shone, and E. Ault, London. Improvements in the purification of sewage and other foul liquids, and in apparatus employed therefor. September 20
- 12653 F. M. Lyte, London. Improvements in the manufacture of anti-fouling paints for submerged structures. September 20
- 12666 W. Edge, London. A new or improved method of wrapping or parcelling up soluble or insoluble blue or other colour. September 22
- 12674 J. Douglas Carter, London. Improved waterproof paper for use in letter-copying. September 22
- 12681 C. E. Bell, London. Improvements in the treatment of oils and tars obtained from blast furnaces and gas producers for the production of valuable hydrocarbons therefrom. September 22
- 12687 J. Riley, Glasgow. Improvements in the process and apparatus for making steel with open-hearth furnaces. September 23
- 12690 W. Kneen, Barrow-in-Furness. The consumption of smoke and economical combustion of coal. September 23
- 12691 J. A. Crocker, London. Improvements in filtering machines. Complete specification. September 23
- 12697 F. Oppenheim, London. An unctuous, siccative and emollient mixture for toilet and medicinal purposes. Communicated by E. Dantrevaux, Paris. September 23
- 12699 J. Wyburd Furrell, London. A new or improved method of and apparatus for precipitating or abstracting water from atmospheric air. September 23
- 12702 W. Rudd Oswald, London, and J. Russell Williams, Brockley, Kent. A new metallic, fibrous, and elastic jointing material. September 23
- 12708 S. Cleland Davidson, London. Improvements in apparatus for employing heated air in drying or baking vegetable or other substances. July 29. Previously included in No. 10725
- 12711 A. C. Henderson, London. Improvements in smoke and gas consuming furnaces. Communicated by M. Krudewig, Frankfurt-on-Main, and W. Kloh and J. Hyoes, United States. Complete specification. September 23
- 12713 W. Gostwyck Gard, London. The manufacture of an improved disinfectant, deodorizer and precipitant. September 23
- 12718 H. Brunner, Liverpool. Improvements in the treatment of certain phosphatic materials. September 23
- 12725 E. Marchese, London. Process for electrolytic extraction of copper from its ores. September 23
- 12732 R. Townsend and A. Watson Townsend, Glasgow. Improvements in bleaching fibrous materials and fabrics, and paper pulp of vegetable origin. Complete specification. September 24
- 12741 R. Kennedy, Glasgow. Improvements in the expansion of air or gases by heating. September 24
- 12755 H. L. Pattinson, Junr., London. Improvements in the manufacture of hydrates of barium and strontium from their ores. September 24
- 12765 W. Day, London. Improvements in kilns for burning bricks and other articles. September 24
- 12767 W. Deacon Curzon and G. Jones, London. Improvements in the manufacture of pigments. September 24
- 12793 H. Campbell, London. A new or improved compound applicable for welding iron and steel, purifying and strengthening cast iron, restoring burnt steel, and imparting to Bessemer steel qualities which enable it to be tempered like cast steel. Communicated by W. W. Ker, United States. September 25
- 12803 E. Robbins, London. The manufacture of new compound chemical cements and concrete formations thereof for building and other purposes. September 25
- 12806 J. Inray, London. Process for converting petroleum and similar hydrocarbons into acids, soaps, and compounds of resin and ether kind. Communicated by Dr. E. Schaal, Stuttgart. September 25
- 12807 J. Inray, London. Process for the production of the acid ethers of resinous gums, such as copal and the like. Communicated by Dr. E. Schaal, Stuttgart. September 25
- 12809 E. Biedermann and E. W. Harvey, London. Means of utilising waste gaseous products of combustion. September 25
- 12830 J. W. Slater, London. Improvements in the preparation of agents to be used in the treatment of sewage and other liquid or semi-liquid putrescent or putrescible matters and improvements in the treatment of such matters. September 26
- 12834 W. A. Rowell, London. The manufacture of chromic acid or trioxide of chromium. September 26
- 12841 C. Harrison, London. An improvement in the manufacture of soap. September 26
- 12869 R. Townsend and A. Watson Townsend, Glasgow. Improvements in softening, batching, or lubricating fibrous materials of animal or vegetable origin. September 27
- 12876 C. Cheswright, London. Improvements in laminating metals, and in the apparatus to be employed therein. Partly communicated by H. Cheswright, Bordeaux. September 27
- 12877 H. W. Winshurst, Anerley, Surrey. An improved system for manufacturing a cheap illuminating and heating gas. September 27
- 12891 J. Wyburd Furrell, London. New or improved combined apparatus for precipitating water from atmospheric air and distilling sea water. September 27
- 12893 H. J. Haddon, Westminster. Improvements in drying kilns. Communicated by P. Colparet, Audenarde. September 27
- 12901 J. Budd, London. An improved method or process of imparting to glass the appearance of marble or other stone. September 27
- 12902 J. Budd, London. An improved method of ornamenting or decorating glass in imitation of mosaic or similar work. September 27
- 12912 J. Hislop, Glasgow. Improvements in and connected with retorts for obtaining from coal or other carbonaceous substances illuminating or heating gas, oil, coke, and other products. Complete specification. September 29
- 12918 E. Johnson, Westminster. Improvements in carbonising retorts for the manufacture of filtering and deodorising compounds and for other purposes. September 29
- 12926 S. Goldmann, Dresden, Germany. An improved process for the manufacture of a tooth tincture or tooth wash for effectually curing tooth-ache, so called "Kaiser Zahnwasser." Complete specification. September 29
- 12930 G. W. Moon, London. The amalgamation of gold and silver with quicksilver. September 29
- 12939 J. G. Willans, West Hampstead, Middlesex. Improvements in the manufacture of aluminium and its alloys. September 29
- 12963 J. E. Chapman, Hull, Yorkshire. Oil press wrappers or envelopes used in extracting oil from seeds in cake making. September 30
- 12977 H. J. Worssam, London. A new or improved machine for continuously expressing or squeezing liquids from hops and other materials. September 30
- 12978 W. E. Bovill, London. An improved filtering material. September 30
- 12979 A. W. Gerrard, London. Estimating nitrogen and other gases, and called a nitrogen apparatus. September 30
- 12990 J. Inray, London. Apparatus for combustion of liquid fuel. Communicated by H. de Bay and C. de Rosetti, Cairo. September 30
- 13025 J. Taylor, Cheltenham. The preparation of chlorine gas on the large scale. October 1
- 13029 W. A. and C. A. Biddell, London. Manufacture and treating of iron and steel, and the coating of such, and introducing of vitreous and other matter. October 1
- 13013 J. Swallow, London. Improvements in the preparation of mineral oils for woollen manufacturing and other purposes. October 1
- 13015 J. H. Johnson, London. Improvements in the manufacture of sulphide of zinc, and the obtaining of sulphuretted hydrogen and sulphurous acid as products of the said manufacture. Communicated by T. Macfarlane, Canada. October 1
- 13019 G. Nurse, London. An improved method of finishing terre and tin plates. October 1
- 13059 W. Scott, London. Improvements in the treatment of sewage. October 2
- 13074 J. Simpson and E. W. Parnell, Liverpool. Improvements in obtaining gold and silver from auriferous and argentiferous sulphide of antimony ores. Complete specification. October 2
- 13080 W. H. Samson, London. Improvements in deodorising refuse substances for manures. October 2
- 13105 J. Baynes Thompson, London. Improvements in bleaching. October 2
- 13109 G. A. Sweetser, London. An improved combustible composition to be employed in the manufacture of cigar lights or fuses. October 2
- 13111 F. M. Eppley, London. An improvement in the manufacture of steel. Communicated by J. E. Sherman, United States. October 2
- 13119 F. Mason and J. Conqueror, Sunderland. The manufacture of rolled plate glass. October 3
- 13134 P. M. Justice, London. An improvement in the manufacture of steel. Communicated by L. E. Thomas, Paris. October 3
- 13139 A. C. Henderson, London. Improvements in dyeing and printing animal or vegetable tissues and paper with colours derived from artificial alkaloids by electrolysis. Communicated by L. Aubert, Lyons. October 3
- 13188 W. D. Cliff, London. Improvements in the manufacture of clay retorts. Complete specification. October 4
- 13189 G. Gilders, London. New or improved manufacture of manure. October 4
- 13201 S. Fox and J. Whitley, London. Improvements in the casting of metals. October 4
- 13209 S. Perkins and W. Smellie, Manchester. Improvements in the construction of heating and puddling furnaces used in the manufacture of iron and steel. October 6
- 13217 J. Fyfe and J. Terry, Glasgow. Improvements in compositions for burning and disinfecting. October 6
- 13223 B. F. Elderton, London. Improvements in the treatment of coal gas and apparatus for increasing its brilliancy for illuminating purposes. October 6
- 13235 T. H. Johns and W. A. Johns, London. An improved method of tinning plates. March 25. Previously included in No. 4637
- 13237 J. W. Matteson, W. Ingram Chapman, and T. Goulton Matteson, London. Improvements in the manufacture of Portland cement. Complete specification. October 6
- 13243 Sherwood and Co., London. Improvements in the manufacture of tinted or coloured rolled cathedral glass. October 6
- 13244 A. T. Booth, London. Improvements in apparatus for condensing steam and supplying air to furnaces. October 6
- 13245 C. S. Cross, London. The disinfection and deodorisation of the sewage of towns. October 6
- 13251 J. M. Hale, London. Improvements in the manufacture of non-alcoholic beverages. October 6
- 13256 H. H. Lake, London. Improvements in the manufacture of paper, and in machinery or apparatus therefor. Communicated by W. J. Foley, United States. October 6
- 13258 W. H. Beck, London. A new or improved process for cold scouring and dyeing cotton. Communicated by La Société Ch. Toussant et Cie., Paris. October 6
- 13260 H. J. Allison, London. Improvements in brick kilns. Communicated by S. Wood Underhill and G. Everard Fisher United States. Complete specification. October 7



- 13324 J. Gilligan, Abertillery, Monmouthshire. Improvements in the manufacture of iron. October 7
- 13327 W. P. Thompson, Liverpool. Improvements in hand grates for fire-extinguishing purposes. Communicated by H. Dean Harden, United States. Complete specification. October 7
- 13328 A. T. Smith, Barkingside, Essex. The manufacturing of a material in a granulated form for the purpose of filtering sewage and other fluid matter. October 7
- 13329 N. E. Mattass, London. Improvements in the separation of tin from tin plate. Communicated by P. G. Pellegrini Tibaldi, Milan. October 7
- 13332 E. C. Cortis Stanford, Glasgow. Improvements in compositions or coatings to act as non-conductors of heat. October 8
- 13327 W. D. Curzon and G. Jones, London. Improvements in the treatment of sewage and manufacture of manure therefrom. October 8
- 13337 J. Robbins, London. The preparation of silicates with suitable neutral materials for the purpose of rendering them applicable for use in cements and other compounds which are required to set or harden. October 8
- 13343 W. R. Lake, London. Improvements in the manufacture of medicated paper, and in apparatus therefor. Communicated by S. Wheeler, United States. October 8
- 13350 J. W. Chaik Webb, Worcester. Improvements in kilns and stoves. October 9
- 13355 W. Smith, Dublin. The manufacture of waterproof cement. October 9
- 13358 W. Hibbert, Manchester. An improved mixture or compound for preventing or arresting the decomposition and putrefaction of animal and vegetable matters. Complete specification. October 9
- 13368 J. Hanson, London. Improvements in the treatment of town refuse matters, and in the manufacture of manures therefrom. October 9
- 13380 J. J. Barclay, R. Allison, and J. Barclay, London. Improvements in kilns and apparatus for drying and burning bricks. October 9
- 13425 C. D. Abel, London. The production of articles of metal (plain or enamelled) glass, porcelain, ceramic ware, pottery, fayence, and natural and artificial stones with a coating or ornamentation of aluminium or aluminium bronze. Communicated by Dr. G. Gehring, Landshut. Complete specification. October 10
- 13426 T. Nordenfelt, London. Improvements in furnaces for melting metals in crucibles. Communicated by C. G. Wittenstrom, Motala, E. Faustman and P. Ostberg, Stockholm. October 10
- 13433 E. C. Cortis Stanford, Glasgow. Improvements in the manufacture of algin and other useful products from seaweeds. October 11
- 13443 R. W. Thom, Liverpool. Improvements in apparatus for washing, scrubbing, and purifying, and also for charging and impregnating coal and like gases with hydrocarbon vapours. October 11
- 13446 G. T. Baily, Glasgow. Purifying paraffin oils. October 11
- 13460 T. Vosper and J. Eastcott, London. An improved apparatus for washing, cleansing, and separating metallic ores, particularly adapted for the ores of tin, lead, and gold. October 11
- 13475 G. G. André, London. Improvements in explosive compounds. October 11
- 13476 G. G. André, London. Improvements in explosive compounds. Complete specification. October 11
- 13489 R. Bartlett, London. A testing apparatus for liquids boiling in vacuo. October 11
- 13492 A. French and J. Ballantyne Hannay, Glasgow. Improvements in making white and other lead pigments, and in apparatus therefor. October 13
- 13491 H. J. Rogers, Watford. Improvements in apparatus for the manufacture of gas. October 13
- 13501 J. Smith, London. A process of treating manilla and other fibres for the purpose of extracting the resinous, gummy, and colouring matters therefrom. October 13
- 13512 J. Seeger, London. Improvements in lubricating oils. October 13
- 13522 W. E. Gedge, London. Improvements in the manufacture of blasting powder from nitrated cellulose. Communicated by W. F. Wolff, Walsrode, and M. von Forster, Berlin. Complete specification. October 13
- 13518 W. Charleson, Manchester. Improvements in varnish or composition for coating ointment boxes and other articles and bodies. October 11
- 13553 B. Hammond, Guildford, Surrey. Improvements in the manufacture of soap. October 11
- 13560 A. Gutensohn, London. An improved process for coating iron or other metals with tin, lead, or terne. October 11
- 13561 H. P. Howard, S. Jones, and F. Jones, London. Improved printed waterproof paper. October 11
- 13579 H. H. Lake, London. An improved method or process of and apparatus for recovering paraffine, stearine, or similar wax-like substances from petroleum or other oil. Communicated by R. H. Smith, United States. October 11
- 13580 H. H. Lake, London. Improvements relating to the extraction of paraffine or other solid substance from oil or other liquid, and apparatus therefor. Communicated by R. H. Smith, United States. October 11
- 13585 E. Capitaine, London. Purification of waters. Communicated by M. Nahnsen, Schönebeck a. Elbe. October 11
- 13590 P. Jensen, London. Improvements in the production of alkali carbonates, and in apparatus therefor. Communicated by A. Vogt and A. Figge, Westerhansen. Complete specification. October 11
- 13593 W. Hassall, London. Improvements in fire-bricks or furnaces for heating pottery, brick, and like kilns or ovens. Complete specification. October 11
- 13629 H. W. Fenner, London. Improvements in the distillation of coal tar and other hydrocarbon substances, and in the apparatus employed therein. October 15
- 13630 H. W. Fenner, London. Improvements in the heating of stills or boilers employed in the distillation of coal tar and other hydrocarbon substances. October 15
- 13650 B. P. Walker, Birmingham. An improved method of heating substances for the purpose of distillation. October 16
- 133067 A. J. Bickmore, London. A new or improved process of manufacturing from cereals and pulse an extract containing nutritious value of the seeds without the coarse fibres, the extract being gained from the bran and similar covering, and said extract being for use in bakers' and confectioners' articles, soups, and so forth. Communicated by H. Lehrenkraus, Stuttgart. October 16
- 13678 C. F. Cross and J. P. Rickman, London. Improvements in bleaching. October 16
- 13722 G. L. Wigg, M. Steele, and W. J. Wigg, Liverpool. Improvements in the treatment of certain residual liquors to obtain useful products therefrom. October 17
- 13725 J. J. Coleman, Glasgow. Improvements in preserving animal or vegetable substances. October 17
- 13726 E. J. Mills, Glasgow. Improvements in the treatment of alcoholic liquids. October 17
- 13711 P. Moir Crane and P. Holland, London. Improvements in the distillation of hydrocarbon oils. October 17
- 13745 J. Humphrys, London. A new or improved method of and apparatus for melting zinc and other metals. October 17
- 13753 S. Smille, London. Improvements in apparatus for heating and cooling fluids. October 17
- 13761 M. Dennstedt, London. Improvements in and relating to the manufacture and preservation of articles of gypsum stucco, or like. October 17
- 13784 J. H. Riley and J. Downham, Bury, Lancashire. Improvements in means for preventing the destruction of cotton selvages in all kinds of woollen piece goods by the acids, etc., used in the process of extracting vegetable matter therefrom. Complete specification. October 18
- 13800 W. Hope and G. F. Deacon, London. Improvements in and apparatus for turning over and exposing grain, salt, drugs, cement, and other substances and cereals and materials for the purpose of drying, cooling, oxidising, or otherwise treating. October 18
- 13815 W. H. Beck, London. A new or improved process for obtaining useful products from the yolk or grease of wool. Communicated by C. Viollette and A. Buisine, Lille, and A. Vinchen, Roubaix. October 18
- 13818 A. Angell, London. Improvements in the manufacture of materials for use in the purification by filtration of sewage and other foul liquids and water, and in the purification of gases and in the decoloration of sugar and other matters, and for other like purposes. October 18
- 13833 L. Perroncel and J. Grathwohl, London. The employment of asbestos, or other incombustible substance, in the manufacture of portfolios or portable cases, so as to protect them against destruction by fire. October 20
- 13867 A. J. Bonit, London. Improvements in the manufacture of paint. Communicated by S. Bond, Constantinople. October 20
- LIST of DISCLAIMERS and MEMORANDA of ALTERATIONS relating to Specifications filed previous to 1st January, 1884, with No. and Date of Patent and Date of Disclaimer.
- 3731 of 1877—H. Caro (Badische Aniline and Soda Fabrik). Colouring matters. March 2, 1883
- 1678 of 1878—F. Windhausen. Manufacture of ice. January 6, 1883
- 1188 of 1878—E. W. Parnell. Manufacture of alkalies. May 31, 1883
- 2010 of 1880—C. Pieper (communicated by F. Windhausen). Manufacture of ice. January 6, 1883
- 4491 of 1881—J. Imray (communicated by La Société Anonyme des Produits Chimiques du Sud-Ouest). Manufacture of soda. November 17, 1882
- 4544 of 1881—E. Turpin. Manufacture, etc., of hypo-nitric anhydride. November 21, 1882—May 10, 1884
- 5024 of 1881—E. Carey, H. Gaskell, junr., and F. Hurter. Manufacture of bicarbonate of soda. February 2, 1883
- 1161 of 1882—E. Turpin. Explosive compounds. April 23, 1884
- ENGLISH PATENTS PUBLISHED.\*
- APPLIED FOR DURING 1883.
- 1161 of 1882 Disclaimer. The Société Anonyme Panclastite (Turpin's Patent). Manufacture of explosive compounds, etc. 1d
- 5355 B. J. B. Mills. Communicated by Manhès. Process for the treatment of sulphurous and arsenical composite ores containing nickel, cobalt, and other metals. 1d
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- 3731 of 1877—H. Caro (Badische Aniline and Soda Fabrik).  
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 6, 1883  
 4188 of 1878—E. W. Parnell. Manufacture of alkalies. May  
 31, 1883  
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 anhydride. November 21, 1882—May 10, 1884  
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275 W. G. Little. Compounds for the destruction of insects upon vegetation, etc. 2d

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11191 O. Imray. Communicated by Harris. Fertilisers. 4d

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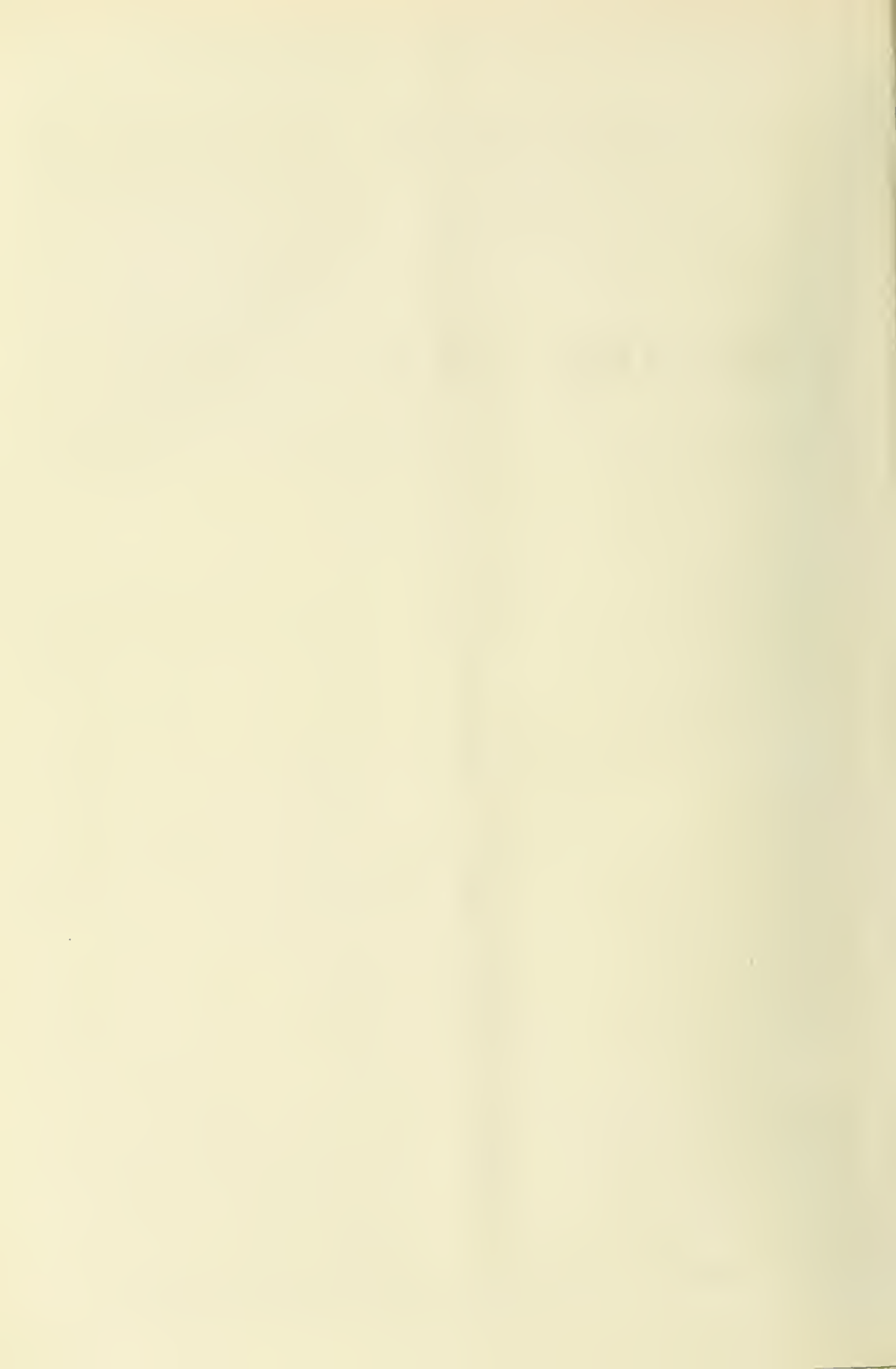
11347 C. Steffen. Producing white crystalline sugar from the raw sugar mass. 4d

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# THE JOURNAL OF THE Society of Chemical Industry:

## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 11.—VOL. III.

NOVEMBER 29, 1884.

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### NOTICES.

The Council of the Society, at their last meeting, had under consideration the advertisement of Mr. G. E. Davis, late Alkali Inspector, which appears in the Journal of the Society. The Council passed unanimously a resolution disapproving of the terms of the said advertisement, but regrets that it has no power to order its rejection owing to a contract with the printers, which is binding until the 31st December, 1884.

In reply to numerous inquiries the Secretary begs to inform Members that the first six numbers of the Society Journal for 1882 have now been reprinted, and can be obtained on application, while the orders already received are being executed with every dispatch. For the information of those desiring to have their copies bound, it may be convenient to know that the index and title page of Vol. I. are to be found in the July number, 1883.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 43, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

Members are requested to take notice that Mr. Ludwig Mond, Honorary Foreign Secretary of the Society, has removed from Winnington Hall, Northwich, to 28, Park Crescent, Portland Place, W.

#### LIST OF MEMBERS ELECTED NOV. 21st, 1884.

C. W. Bickerton, The Hollies, Bosden, near Stockport, colliery agent.  
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Geo. Booth, Irk Vale Dye Works, Middleton, near Manchester, yarn dyer.



Ian Cameron, Nickel Co., Kirkintilloch, N.B., chemist.  
 J. H. Clemes, c/o Henry Clemes, Falmouth, Cornwall, civil engineer.  
 Thos. Crabtree, Spring Valley Dye Works, Failsforth, near Manchester, manager.  
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 Henry Wardale, 52, Bewick Road, Gateshead-on-Tyne, engineer.

#### CHANGES OF ADDRESS.

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 T. Barrrell Bewick, Haydon Bridge, Northumberland.  
 Geo. Bray, Blackman (not Blackburn) Lane, Leeds.  
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 G. Chaloner (late of Islington), 35, King's Road, Green Lanes, London, N.  
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 R. Picknp (late of Baxenden), Bank Mill Printworks, Tonge, Middleton, Lancashire.  
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 S. P. Wilding (late of Rood Lane), 1, Queen Victoria Street, Mansion House, E.C.

#### Deaths.

G. H. Herbert, 7, Akeuside Hill, Newcastle-on-Tyne.  
 G. W. Wigner, Plough Court, Lombard Street.

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MEETINGS, SESSION 1884-5—First Monday in each month (unless otherwise indicated).

December 1st, Mr. J. M. Thomson, F.I.C., F.C.S., will read a Paper "On Photography, for those engaged in Industrial pursuits."

Meeting held Nov. 3rd, 1884.

MR. DAVID HOWARD IN THE CHAIR

The CHAIRMAN said he did not propose to take up the time of the meeting with an inaugural address, as he thought that they had more interesting things to listen to. At the same time he might be allowed to call the attention of all the members of the Society who had not already seen it, to the anniversary address of their president, as President of the Chemical Society, upon the subject of chemical education. It was so admirable and so exhaustive an explanation of the whole subject, and was written with so much thought and experience, that he could only say that—although it was a subject to which he had often devoted his thoughts—when he heard that address, he felt that all that he could have said on that subject was most thoroughly forestalled. Of course it was a subject in which they all felt more or less interested, even those who might imagine that their own education was finished, though he could hardly think that any one engaged in the work of chemistry would be under that delusion. As far as regarded workmen in practical chemistry, he did not think they wanted much technical education beyond that which their employers could give them. The only school for workmen was the workshop. On the other hand, as to the principals, or those who had to find the head work, he was convinced that no amount of training was too thorough and no amount of scientific knowledge was wasted, and that more than ever an increasingly thorough and accurate knowledge, both of general chemistry, and also of the specific subjects which they carried into practice was required of each of them. He could only add one caution. There was great danger now-a-days of over-estimating mere technical instruction; that was to say, of supposing that education consists of learning the specific things required in practice, rather than of the knowledge how to think. He more and more believed that what was required in training a chemist, or any one else, was to teach the process and method of thought rather than specific knowledge. An education confined to what is needed in daily practice may make a scientist—that dread word, bad Latin, worse Greek—but never a scientific man.

ON SOME POINTS IN THE EXAMINATION OF TALLOW AND SOME COMMERCIAL OILS, ESPECIALLY FOR ACIDITY.

BY W. H. DEERING, F.I.C., F.C.S.

The chemical examination of oils and fats is now being so industriously worked at, that I take this

\* Mr. Foster and Mr. North desire to become members as from January 1st, 1885.

opportunity of putting together some results collected during the last eighteen months. Part were obtained in the ordinary course of official business, and I am indebted to my chief, Sir Frederick Abel, for his readily-given permission to make use of these.

Hausmann, in the year 1881, appears to have been the first to have called attention to the practicability of estimating, in an alcoholic solution, free fatty acids in presence of glycerides, by means of phenolphthalein; he used his method for the examination of some of the products of the candle manufacture. Gröger, in 1882, examined Hausmann's method, and showed its precision by test-analyses of artificial mixtures of free fatty acids and fat. E. Valenta, in 1883, gave a most valuable table of KHO required for total saponification of 21 different kinds of oil and fat, but no direct estimations of free acid, though to some extent the acidity can be inferred from the equivalent he gives for the total fatty acids (insoluble in water, however) of nine oils. Valenta's paper is, in small compass, the record of much labour. While his list contains several out-of-the-way oils, one is surprised not to find linseed or sperm oil in it.

Köttstorfer gives no numbers for free acid in his well-known paper in the year 1879.

L. Archbutt ("The Analyst," Sept., 1884) has estimated the free acid in a large number of samples of olive and rape oil, and in some palm oils.

The foregoing, and such results as those of the Messrs. Stoddart, in Allen's "Commercial Organic Analysis," constitute a large body of work, and my results will only furnish some direct estimations of the acidity, and of the equivalent, of some assorted tallows, and of some oils, for which there are not too many numbers on record.

I will give first the numbers actually obtained, and then some experiments on the method used.

The percentages of "KHO required" are parts by weight, KHO per 100 parts by weight of substance.

The "acidity" KHO will include that required for any mineral acid which might possibly be there; the true equivalent of the free acids would be a troublesome number to obtain; the numbers used in some of the above-mentioned papers, to calculate free acid from the KHO required for neutralisation, have in all cases been determined on acids insoluble in water, and these sometimes obtained really from the free acids, sometimes from the total insoluble acids of the oil or fat, *i. e.*, from free acids and glycerides all together. It will, therefore, be safer to give the percentage of KHO.

*Tallow.*—Each sample is from a different source.

Samples of year 1883.	KHO required to neutralise free acid.	Total KHO required for saponification.
No. 1, Russian	2.19%; 2.28%	19.80%; 19.65%
No. 2, Russian	0.51%; 0.56%	19.44%; 19.43%
No. 3, Russian	1.99%; 1.95%	19.78%; 19.66%
No. 4, Russian	2.12%; 2.46%	
No. 5, Russian	1.09%; 1.10%	19.37%; 19.51%
No. 6, Australian Beef	0.35%;	19.32%
No. 7, Australian Mutton	1.43%;	19.58%
No. 8, Town Tallow	1.42%; 1.35%	20.69%; 20.47%
Samples of year 1884.	KHO required to neutralise free acid.	
No. 9, Russian		1.01%
No. 10, Russian		1.24%
No. 11, Doubtful		0.60%; 0.61%
No. 12, Australian (?)		0.42%
No. 13, Australian Mutton		0.78%
No. 14, Town Tallow		0.90%; 0.92%
No. 15, Town Tallow, 6 years old		5.0%
No. 16, New P.Y.C. (Russian)	{ Cask 1, 0.47%; 0.43%	
	{ Cask 2, 0.44%	
	{ Cask 3, 0.41%	
No. 17, Old P.Y.C.		1.01%
No. 18, do.		0.44%
No. 19, do.		0.96%; 0.91%
No. 20, Australian Mutton		0.47%
No. 21, do.		1.04%; 1.09%
No. 22, do.		0.17%
No. 23, Australian Beef		30.47%
No. 24, do.		0.99%
No. 25, do.		1.77%

Samples Nos. 1 to 8 contained no amount of water, mineral matter or organic dirt, capable of affecting the percentage numbers. The others not specially examined for these, but the KHO numbers could not have been affected thereby.

The KHO-acidity percentages  $\times 5$  will give a number probably not far from the truth for the amount of free acids present in these tallows; the mean of the molecular weights of stearic, palmitic, and oleic acids being 274. Using 5 as a factor, and omitting No. 15, the 24 samples would show a minimum of 0.85% free acids, and a maximum of 12%. No. 15, which had been kept in store for 6 years, would contain 25% free fatty acids. To judge from these few numbers, the user of tallow as a lubricant has no certainty of superiority in the matter of acidity when he buys Russian tallow at so much higher a price than Australian mutton tallow.

All the oils examined were refined oils.

*Olive Oil.*—The number of instances given by Mr. Archbutt is so large, that I will only give the acidity of some few samples examined. The minimum number for acidity in seven samples of olive oil was 0.86% KHO, the maximum 1.64% KHO, the mean of the seven 1.28% KHO. If one may be permitted to regard the free acid as equally consisting of oleic and palmitic acids, the factor will be 4.8, and the calculated percentages of free fatty acids will be:—minimum 4.1%, maximum 7.9%, mean 6.1%.

Three samples of Lucca oil required respectively 0.58%, 0.82%, and 0.62% of KHO to neutralise their acidity. Another Lucca required 4.15% (repeat) 4.28% of KHO, or 20.2% of calculated free fatty acids. The estimation of total KHO required for saponification will not be of use in ascertaining the genuineness of olive oil, as the saponification-potash numbers given by the probable adulterants are too near that of olive oil itself.

*Rape Oil.*—The numbers obtained some months since in examining five rape oils intended for lighting, may be of interest. Mineral oil was looked for in Nos. 1, 2, and 4, and its absence shown. All five were examined in other ways, and Nos. 1 and 2 had the characteristics of genuine rape oil, No. 3 of an oil of doubtful quality, and Nos. 4 and 5 of adulterated rape oil. The following numbers were obtained with them:—

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
KHO required to neutralise free acids....	0.21%	0.47%	0.37%	0.78%	0.51%
Total KHO required for saponification ..	17.08%	17.30%	17.31%	17.55%	17.52%

The equivalent of oil No. 1 is 328.6, of No. 2 is 324.4 where KHO=56.13 : 7 other rape oils, not specially burning oils, required an average of 0.62% KHO to neutralise their acidity, the minimum being 0.42% and the maximum 1.31% KHO. Omitting the maximum, the remaining 6 give an average of 0.51% KHO.

*Cotton Seed Oil.*—Three different oils examined:—

	No. 1.	No. 2.	No. 3.
KHO required to neutralise free acid..	0.11%	0.03%	0.13%
Total KHO required for saponification	19.20%	19.66%	19.52%
Equivalent of No. 1 is 292.			

*Raw Linseed Oil.*—As few saponification numbers are recorded, and so far as I know no acidity numbers, I give five oils in full. All five were examined for unsaponifiable oil; none in any.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
KHO required to neutralise free acid .....	0.24%	0.25%	0.15%	0.24%	0.32%
Total KHO required for saponification .....	18.82%	18.97%	18.80%	18.74%	19.12%

Another sample, less likely to be good, required 1.16% KHO to neutralise free acid.

*Boiled Linseed Oil.*—Two samples examined for acidity. No. 1 required 0.93% KHO to neutralise free acid; No. 2 required 1.59%. Nos. 1 to 5 of the raw



oils give as a mean 18.89% KHO for saponification, the equivalent therefore will be 297, where KHO = 56.13.

It is rather surprising, in such a drying oil as linseed, to find such small amounts of free acid as in the 5 raw oils. The money inducement to adulterate linseed oil, one would think not to be very great; but if the oil's acidity be low, common rosin cannot be present in any quantity, as in one experiment I found that 17.035 grms. of KHO were required to neutralise 100 of rosin. The equivalent of that rosin would be 329.5 (Hegner in his paper on "Bees' Wax" gives 329 as the equivalent), and each 1 per cent. rosin would throw up the "acidity" KHO of an oil about 0.17%.

The dried fatty acids (insoluble in water) from one sample of raw linseed oil I found to require 20.33 grms. KHO per 100 of acids (equivalent 276). Valenta gives 20.33% KHO for the acids of cotton oil. If an extended examination of the water-insoluble fatty acids from the oils and fats used for soap (and especially soft soap) should show that all pretty nearly require 20% KHO for neutralisation, the difference between that number and that given by rosin might be a useful, indirect means of estimating the rosin in soap, by titrating the separated fatty acids. For instance, the water-insoluble fatty acids from a soft soap which had an odour of rosin, required 19.41 grms. KHO per 100 of acids. The solubility of the potash soap of linseed oil is remarkable, and is perhaps a characteristic of other drying oils. In nine cases I have found the soap from linseed oils quite fluid in the cold; when under the same circumstances, the soap from rape oil forms a jelly. 40 grms. of oil had been saponified and made up with water to about 800 cc., an excess of about 10 grms. of KHO being also in the solution.

Castor Oil.	No. 1.	No. 2.
KHO required to neutralise free acid.....	0.22%	0.19%
Total KHO required for saponification.....	18.02%	
Equivalent of No. 1 is 312.		

Sperm Oil.	KHO required to neutralise free acid.	Total KHO required for saponification.	Equivalent.
No. 1. Best quality. "Cold bagged".....	0.29%	13.36%	
" 2. Seconds. "Hot pressed".....	0.42%	11.23%	
" 3. Probably of intermediate quality.....	0.15%	13.77%	
" 4. A good oil.....	0.42%	13.43%	
" 5. Of doubtful quality.....	0.24%	12.34%	
" 6. ".....	0.11%	12.51%	
" 7. A bad oil.....	0.41%	11.74%	
Bottle-nose whale oil.....	0.42%	12.60%	
The equivalent of oil No. 1 is 420, of No. 4 is 418, of bottle-nose oil is 416.			

Seal Oil.	KHO required to neutralise free acid.	Total KHO required for saponification.	Equivalent.
No. 1.....	1.43%	19.08%	294.
" 2.....	0.41%	18.90%	297.
Southern Whale Oil.....	1.51%	19.31%	291.

From these numbers, and the ready saponifiability, seal and whale are, no doubt, preponderatingly glycerides.

*Myrtle Wax.*—Although somewhat outside the subject of these notes, I give the result of one titration of this substance, as it is one not included in Hegner's paper on bees' wax; which latter, there is reason to believe, it is used to adulterate, its price being low compared with that of bees' wax. The sample of the commercial article examined was a hard, pale green, solid. It contained very little dirt or extraneous matter. It was very readily saponified, and gave these numbers:—

KHO required to neutralise free acid.....	0.44%
Total KHO for saponification.....	20.57%
Equivalent 272.9, which is very nearly that of tripalmitin 268.7.	

An old statement by Moore is to the effect that myrtle wax contains a large quantity of palmitic, and a small quantity of myristic acid, for the most part in the free state. It must have been a very different material to the one I examined. I should be glad to have the opportunity of more completely examining some more samples.

*The method of titration examined.*—Subjoined are the results of experiments on the method followed. Simple as the method is, there are some points which it is well to settle once for all.

I always use strong rectified spirit of wine (of about 0.82—0.83 sp. gr.) in preference to methylated spirit, both as solvent for the fatty acids and for the alcoholic potash solution. The homogeneity of good spirit of wine is a great advantage, and the alcohol can be recovered and dehydrated.

I use half normal alcoholic potash, phenol phthalein as indicator, and 5 to 6 grms. of fat or oil, when only acidity is to be estimated. To the fat or oil in a suitable flask, 50 cc. alcohol is added, and boiled for 3 minutes in water-bath, the flask being attached to an inverted condenser, and shaken round once or twice while in the bath. A little phenol phthalein added, and the potash solution dropped into the liquid, while the flask is kept rotating. The purple colour is bleached instantaneously, until neutrality is reached, when the purple colour produced by a slight excess of potash fades with sufficient slowness as to leave, in most cases, no doubt as to the point at which to stop. As was to be expected, the colour does not last as well in presence of excess of glycerides as in the corresponding case with bees' wax; but except in the case of excessively acid oils and tallows, and of some old P.Y.C. tallows not excessively acid, the colour stands well enough to hit the point with sharpness. In the cases referred to of excessive acidity, the colour often fades rapidly, the grave decomposition which has taken place apparently leaving the non-acid portion readily open to saponification. Still, even in these cases, a fair result can be obtained. Some old P.Y.C. tallows give a yellow colour as neutrality approaches, but this does not interfere with the purple colour.

One point of gravity has to be settled, viz., Is the free acid produced by boiling the glycerides with the alcohol? It is conceivable that the alcohol might cause a slight hydration of the glycerides, the acid set free not being converted into ethyl ether at the rate of glyceride decomposition. This I have endeavoured to answer by comparative experiments with tallow, and with some of the principal oils, which were boiled with alcohol for three minutes and for one hour; and in the case of the oils, by also estimating their acidity in a cold solution of the whole oil. Tallow is not sufficiently soluble in cold ether to be able to make a cold titration with it.

When a few drops of alcoholic phenol phthalein are added to 50 cc. of ether, and a slight excess of alcoholic potash added, the purple colour is produced; but the liquid is slightly opalescent, the potassic phthalein apparently forming an emulsion, and in the course of hours going out of solution. I used for the cold solution of the oils 50 cc. ether + 25 cc. alcohol, the mixture neutralised (the phenol phthalein having been added) with a drop or two of alcoholic potash, and added to the oil, and the solution titrated at once in the cold; the reaction went on sharply, and the point was easy to hit, except in the case of the boiled linseed oils which became turbid as neutrality approached. The castor oil was titrated in cold alcoholic solution (without ether). The following numbers were obtained:—

	75cc. Ether-alcohol, in the cold.	50cc. alcohol, boiled for 3.	50cc. alcohol, boiled for 1hr.
Russian tallow.....	—	0.43%	0.47%
Russian tallow.....	—	0.96%	0.91%
Town tallow.....	—	0.92%	0.90%
Australian mutton tallow.....	—	1.04%	1.09%
Australian beef tallow.....	—	0.18%	0.47%
Olive oil.....	1.51%	1.50%	1.50%
Rape oil.....	0.42%	0.43%	0.45%
Raw linseed oil.....	1.12%	1.11%	1.16%
Boiled linseed oil.....	0.96%	0.93%	0.93%
Boiled linseed oil.....	1.64%	1.59%	—
Castor oil.....	50cc. cold alcohol. 0.18%	—	0.21%





so as not to hurt the feelings of the present generation. I was quite prepared for the contention that this was no longer the case, or even that it never had been the case, but I confess I was not prepared for a defence of rule-of-thumbism as a fine art. Indeed, it seems to be more than this: it is a kind of religion, standing in the same relation to rule-of-thumbists that Buddhism does to Buddhists. It is a faith, the simple tenets of which are summed up in its concise creed: "An ounce of practice is worth a ton of theory." Indeed, this convincing formula was actually thrown at my head the other day by a high priest of the cult. Let me set myself right on this score. As a practical man myself, I have never disparaged the value of practice, it must always be the ultimate test of our theories. In former times, when the value of inductive reasoning was almost unknown, facts—most valuable facts—were ascertained. Experiments were tried, not with the object of testing any theory, but simply to see what would come of the operation. In modern times we have gone to work more scientifically. We deal with a certain class of ascertained facts. We construct a theory to explain them. We conclude from our theory that such and such things will happen under such and such circumstances. We try the experiment. If our anticipations are fulfilled, our theory is, to that extent, confirmed. If they are not, we have to modify the theory, so as to make it square with the facts. This modified theory serves as a fresh point of departure for new experiments. So we go on, alternately theorising and experimenting, until we build up a science based on sure foundations. In this way we have built up the science of chemistry, and it is mainly to modern theory, thus corrected and purged from error, that we owe the present development of practical chemical technology, brewing and distilling not excepted. The views I submitted to you last June were not purely theoretical, the views of a mere scientist, as some critics have been pleased to term me. They are the results of theory corrected and chastened by practice, and a practice, as some of you are aware, on a tolerably large scale. I have called rule-of-thumbists those who, adhering to old traditions, resent the intrusion of the scientific worker; who deride his reasoning, based upon honest experimental work, simply because the theory does not happen to square with their own preconceived notions, and perhaps also because they are unable to perceive the immediate practical bearing of it. This is not the way in which progress in any industry is achieved. Let theory and practice go hand in hand. The one is powerless without the other. It is certain that no industry, however great or however ancient, has ever suffered from the light thrown on the true nature of its operations by the labours of the scientific worker.

My paper was intended to be a short summary of our present knowledge of the subject, and I was obliged to allude to many matters in the briefest possible way. Two of these have been brought rather prominently forward in the brewing journals, viz., the influence of electricity on fermentation, and Nägeli's vibratory theory of fermentations. With your permission I will take this opportunity of treating these matters at somewhat greater length.

In my original paper I quoted from Mr. Faulkner's book\* the following passage: "We are apt to have too many metal fittings (in the fermenting vessels), and the circular coils of attemperating piping, with brass parachute pipe passing down the centre line, forcibly reminds one of a Leyden jar arrangement, and in many instances seems to be one." Then,

in a foot-note, he describes a Leyden jar, and says: "In the arrangement spoken of, the parachute pipe would be the inner coat, the attemperating coils the outer, and the layer of beer between them the intervening glass!" and then I put a note of admiration, without further comment. In a letter to the *Brewers' Journal*, of Aug. 15th, Mr. Faulkner maintains the accuracy of his views on electricity in general, and Leyden jars in particular. He goes on to suggest that "Dr. Squire should come and see the Leyden jar arrangement that so amuses him, and explain if he can any other possible solution of the problem that an electrician was requested to investigate." I am not aware," he says, "whether Dr. Squire disbelieves in the idea that electricity has some influence upon the stability and general soundness of beer, or that combinations of dissimilar metals may give rise to galvanic currents, but it is pretty evident that he has no experience of practical brewing," and so forth.

I am surprised that Mr. Faulkner, with all his knowledge of electricity, did not see that in the last sentence quoted he cuts the ground from under his own feet, in fact, smashes his Leyden jar. No one doubts that dissimilar metals plunged into a liquid capable of acting chemically on one of them give rise to a current of electricity, provided always that the liquid is a conductor of electricity, and, moreover, a conductor of electricity of low tension, or rather low electromotive force, as it is now the fashion to call it. I do not deny that beer is such a liquid. *A fortiori*, it is a conductor of electricity of high electromotive force. How, then, can beer represent the intervening glass in Mr. Faulkner's Leyden jar, where it has to act as a dielectric, or non-conductor of electricity of high electromotive force. Has Mr. Faulkner never

heard of Ohm's fundamental formula  $I = \frac{E}{R}$ ? His

beer is a conductor, or a non-conductor, as it suits his purpose. It is more than that, it is like a beam which resists a ton but yields to a pound. So much for the Leyden jar arrangement.

As the notion that electricity has some influence on fermentation is rather widely spread, in England at least (I have not met with it among brewers on the Continent), it may be worth while to go a little into the real facts. For the reasons I have just stated, I could not construct a Leyden jar precisely on Mr. Faulkner's pattern, but I have endeavoured to get as near to it as the laws of electricity would permit. In fact I took an ordinary Leyden jar, closing the mouth airtight. The usual rod, with chain attached, was passed through the bung, which was also furnished with a bent tube, by means of which the evolved carbonic acid gas could be collected and measured. A quantity of wort to which some yeast had been added was divided into three equal parts. One portion was put into this Leyden jar, another was put into a precisely similar jar with an internal metallic coating only, and the third was put into a jar with no metallic coating, either inside or outside. The conditions of temperature were the same in all. The Leyden jar was kept charged by occasionally turning the handle of a small electrical machine. Under these conditions, I was unable to detect the slightest practical difference between the three fermentations. In another experiment, a small Ruhmkorff coil was connected with the Leyden jar, so that it was constantly charged and discharged by placing a brass ball, connected with the outer coating, near the ball connected with the inner coating. The result in this case was the same. Lest it should be said that all three jars were affected by the very thundery weather I was producing in the laboratory

\* "The Theory and Practice of Modern Brewing," by Frank Faulkner, London. F. W. Lyon.

the other two were placed in another room. The direct passage of sparks about one inch long from a Rhunkorff coil through the fermenting liquid also yielded negative results.

An experiment, several times repeated, was made with a battery current of five Bunsen cells arranged in series, the current entered and left the fermenting wort through platinum electrodes, so as not to complicate the conditions by the solution of any metal. Under these circumstances I have been quite unable to detect any disturbance whatever of the fermentation.

In case of bacterial growth the conditions are somewhat different. On this subject Cohn and Mendelsohn\* have made an elaborate series of experiments. They found that the current from a single Bunsen element had practically no effect on bacterial life. With two such elements the liquid was after 12 or 24 hours partially sterilised at the + pole. But in neither case were the bacteria killed. When transferred to fresh liquid they continued to develop, and the liquid thus partially sterilised for bacteria was perfectly able to support the life of mould organisms. A current from five elements, maintained for 24 hours, not only kills the bacteria present, but *permanently* sterilises the liquid, so that new bacteria introduced into it refuse to develop.

I have repeated these experiments, and can confirm their accuracy. The permanent sterilisation of the liquid by a strong current is a very remarkable fact. It is evidently due to the formation of some product of electrolysis, which remains dissolved in the liquid, after the electric current has been interrupted. My experiments lead to the conclusion that this substance is peroxide of hydrogen, which is known to be very inimical to bacterial growth. Indeed, according to Paul Bert and Miguel,  $H_2O_2$  is, of all known substances, the most powerful antiseptic, exceeding, in this respect, bichloride of mercury, which stands next to it in order. I merely put this forward now, provisionally, as a matter of opinion, of the truth of which I hope shortly to be in a position to furnish proofs.

With a mixture of yeast and bacteria I have been unable to obtain permanent sterilisation, owing, as I suspect, to the decomposition by the yeast of the peroxide of hydrogen as soon as formed.

From what has been said, I think we may venture to draw the following conclusions:—

1. That electricity, whether static or dynamic, has no influence whatever on alcoholic fermentation.

2. That electricity, *quid* electricity, has no direct influence on bacterial growth, but if the current is sufficiently strong to cause a perceptible evolution of gas, certain products of electrolysis, probably peroxide of hydrogen, accumulate until the liquid is sterilised.

3. That in practical fermentation when both yeast, and more or less bacteria, are present, electricity produces no effect whatever, either on the yeast or the bacteria.

4. That if dissimilar metals electrically connected are plunged into a fermenting wort, capable of acting chemically on one of them, a salt of that metal *may* pass into solution in consequence of the electrical action. These salts (particularly of certain metals), if present in sufficient quantity, may prejudicially affect the fermentation, but the circumstances under which sufficient quantities of metal would be dissolved are extremely rare.

In the *Brewers' Journal* of Sept. 15th, there appeared an "Occasional Note" on Vibratory Ferments. The author, who evidently knows just so much of the

subject as he could gather from my very short reference to it in my paper, says—

"We note that, at the present time, very advanced scientists are connecting molecular vibration of protoplasm with the fermentive capacity of the yeast cell containing it. This is an entirely novel theory propounded by Nägeli, and he utilizes it to explain what has hitherto seemed inexplicable. Nägeli supposes one form of ferment life to be under the influence of another, and argues that powerful vibration tends to suppress less powerful vibration, and that each ferment has a peculiar vibration of its own, by virtue of which it sets up its own particular decomposing action, and thus we are led to understand how the action of one distinct ferment interferes and prevents the vital movement of any other possessing inferior vibratory power. Here we pause and draw breath, for we are dipping into subjects far beyond the region of pure experimental reasoning, and dealing with theories founded on analogies neither distinct nor convincing."

Leaving the critic, scant of wind, to pause, let us examine how far "pure experimental reasoning" bears on this subject. Nägeli's position is this.\* He points out that it is just those organisms which are incapable of producing fermentation, which are also incapable under any circumstances of living in the absence of free oxygen. Take the various moulds for instance, and even certain kinds of saccharomycetes (*S. Mesentericus*). Those organisms on the other hand which produce fermentation (yeast, *mucor*) are capable of vegetation in the absence of free oxygen, *provided that energy is set free by the decomposition which ensues*.

Assuming that the action and life of these organisms are due to molecular vibration, the energy, necessary to maintain this mechanical movement, may be derived either by means of direct oxidation by free oxygen, or from the energy set free by the decomposition of the substance (sugar) fermented, provided the organism is capable of causing such decomposition. Pasteur's theory is wholly different. He maintains that those organisms which produce fermentation require free oxygen for their respiration. That this is, in fact, the normal condition of their growth. When they are drowned, as it were, in a solution of sugar, they are asphyxiated, and gasping for breath, they turn in desperation to the sugar, which they break up in order to get a supply of that oxygen of which they find themselves temporarily deprived. It follows from this, that the growth of the yeast must always be in a definite relation to the amount of sugar decomposed.

Nägeli's theory does not postulate this. What the yeast requires from the decomposition of the sugar is energy to support the mechanical vibrations of the protoplasm. Of this there is always set free enough and to spare, the balance appearing in the form of heat, which raises the temperature of the fermenting liquid.

The growth of the yeast itself is an affair of sufficiency of the proper food necessary for its development, of temperature, and of neutrality or acidity of the medium in which it grows. As a practical manufacturer of pressed yeast on a very large scale, I am in a position to state that, according to my experience, there is no relation between the quantity of sugar decomposed and either the quality or the quantity of the new yeast produced. These are governed by conditions quite independent of the amount of alcohol obtained, though as might be expected, as a rule, the better the quality of the yeast, the larger will be the crop. According to Nägeli, fermentations of all

\* Beitr. z. Biol. Bd. III. Heft 1.

Nägeli. Theorie der Gärung.



kinds, are produced by the molecular vibration of the protoplasm of the yeast cell. These vibrations are capable of being propagated to a small but sensible distance, in the solution in which the organism finds itself. It is within the spherical space thus set into molecular movement that the decomposition occurs. How far is this idea supported by pure experimental reasoning?

If we take a grape, or other thin skinned fruit, and examine it with a microscope, we shall find it dotted all over with little germs which have been deposited from the air. If we place the grape in water, the organisms become active, and after a little time fermentation is set up inside the grape, and little bubbles of carbonic acid are formed, under the skin, which can be clearly seen by the eye. If now we wipe the grape, and carefully remove the skin, we find that in no single instance are we able to detect underneath the skin the slightest trace of a yeast cell. In one word, the yeast cell has propagated its molecular action through the thickness of the skin to the fermentable juice below. If we measure the thickness of the skin we shall find that this action must have extended to a distance varying from  $\frac{1}{10}$  to  $\frac{1}{50}$  of a millimetre.

Under certain conditions, and this is particularly the case in the fermentation of wines, acetic ether is formed. Now, if we ferment a solution of sugar containing acetic acid, not a trace of acetic ether is produced, nor is acetic ether produced when alcohol is converted into acetic acid, by the action of living cells. But if the two actions are allowed to go on together, if we take a solution of sugar in dilute alcohol, and add to it both the acetic and alcoholic ferments, and provide for the access of air, acetic ether is produced in abundance. It is clear that the acetic acid cannot be produced in the acetic cell, and be converted into acetic ether some time after when it comes in contact with the sugar, while it is being decomposed in a yeast cell as Pasteur's theory requires. The acetic acid and the alcohol must be produced at one and the same time, and at one and the same place, and it is clear that this condition cannot be fulfilled inside either the acetic cell, or the yeast cell, but must take place outside both. If we assume both cells to be surrounded by a zone of molecular vibration, the alcohol will be produced in the zone surrounding the yeast cell, and the acetic acid will be produced in the zone surrounding the acetic cell. If the two cells are sufficiently near to each other these zones will cut. At the point where they overlap, the two actions will be going on at one and the same place, at one and the same time, and acetic ether will be the result.

It has long been known that one kind of fermentation has a tendency to suppress another kind. For instance the lactic ferment develops with difficulty in the presence of an active alcoholic fermentation. If we examine this phenomenon more closely we shall find that the statement is true only under certain circumstances. If to any wort, free from organisms, we add a *small* quantity of yeast and a *small* quantity of lactic ferment, we shall find that the two organisms go on developing quite independently of each other, neither seeming to influence the other. Even if the yeast is present in much larger quantity than the lactic ferment, it does not seem to restrain it in any way, *provided that the total amount of cells present is small as compared with the quantity of wort*. If the solution is neutral, or nearly so, the lactic acid ferment will grow more rapidly than the yeast. If solution is acid the reverse will be the case. It is only after some time, when the cells become numerous, that they interfere with each other. In other words, it is only when the cells, suspended in the liquid, are

so near to one another that the one kind of cell is included within the zone of molecular vibration of the other, that one or the other kind of organism begins to suffer. It is not, as the writer of that "Occasional Note" seems to think, that the vibration of a yeast cell is necessarily more powerful than that of a lactic acid cell, or *vice versa*. It is a question of majority, and like all majorities it tries to stifle the minority. If, when the hand-to-hand fight begins, the yeast is in the majority, it will gain the victory over its antagonist; if it is in the minority, it will have to succumb.

If, instead of starting with a small quantity of organisms, and allowing them to grow till they get to close quarters, we add at once such a quantity of yeast, that when the cells are diffused in the wort, the whole space is filled with the zones of vibration surrounding the cells, then any small quantity of another organism, introduced purposely or accidentally, will have the greatest difficulty in making its way.

It results from careful experiments, that when thoroughly active yeast is present to the extent of one per cent., it completely, or almost completely, prevents the growth of other ferments present in small quantity. It follows then that supposing the yeast to be uniformly diffused through the liquid, each cell would be surrounded by a mass of liquid 100 times larger than itself, through the whole of which its molecular influence may be supposed to extend. The radius of such a sphere would be about twice the diameter of the yeast cell itself. But the yeast is as a rule far from being equally diffused. Some lies at the bottom, some—and generally the greater part—is at the top, so that we may assume that each cell of the yeast, which is equally diffused, is surrounded by a quantity of liquid from 2 to 500 times its own bulk. In calculating the radii of such spheres, we find that the action of the yeast must extend to a distance of from 3 to 4 times its own diameter. As the diameter of a yeast cell is about the 100th part of a millimetre, it follows that the zone of effective molecular vibration must extend to a distance of about 0.025 to 0.035 of a millimetre from the surface of the cell. This agrees fairly well with the measurement 0.020 to 0.050 millimetre obtained by the grape experiment.

I confess I am quite unable to see what other explanation of the restraining action of one ferment on another is possible. It could not possibly be due, as has been suggested, to some substance secreted from the yeast cells itself, for the wash waters of yeast are, without any exception, the very best medium for the growth of bacteria.

I must not trespass longer on your patience, but I have by no means exhausted the arguments in favour of this beautiful, and to my mind the true, theory of fermentation. Enough, I think, has been said to show that, in considering it, we are *not* dipping into subjects far beyond the region of "pure experimental reasoning."

In conclusion, may I be allowed to indulge in a speculation which, I hope, is not an extravagant one. Though I put it forward entirely on my own responsibility, I do so with the greatest diffidence.

We know that bacteria are, in a great many cases at least, first weakened and then ultimately killed by the products of their own action.

We know that many diseases such as erysipelas, gonorrhoea, glanders, and tuberculosis, are due to minute organisms, and there is every reason to expect that many others are due to the same cause.

It is highly probable that the action of these organisms is due to molecular vibration extending to a small but sensible distance, and we have seen that these

vibrations are repressed, and the organism rendered inert in the presence of superior vibration, not in harmonic unison with them. I ask is it not possible that the tissues of the animal body may also be in a state of active molecular vibration, which is able to repress and render inert the bacteria, with the germs of which the air is loaded, and partly through our food, and partly through the lungs, are conveyed into our bodies in countless myriads.

As long as the molecular movement of our tissues is vigorous, we are safe. But when, from excesses of any kind, defective nutrition, or anxiety of mind, this molecular vibration becomes enfeebled, one or other of these tiny organisms gets a footing and begins to multiply. Then comes the struggle for existence. As the bacteria increase, so do they, by their molecular vibration, enfeeble the molecular vibration of the tissues. If this continued, the molecular motion of the tissues would be ultimately suppressed. But, at the same time, the bacteria are giving rise, at any rate in some cases, to products which weaken their own action, and ultimately kill them. At this point the vibratory movement of the tissues, freed from the disturbing action, revives, and the patient recovers. If matters go too far, or if the bacteria present happen to be of a kind not very sensitive to the products of their own action, the molecular vibration of the tissues goes on diminishing until death ensues. Then the molecular movement stops altogether. Freed from this control, bacteria of all kinds have free play, and the dead body swarms with life. I am convinced that if ever we discover any approach to the "elixir vite," if ever our investigations lead to a prolongation of human life beyond its present average span, the road to that discovery lies through the study of the infinitely small.

It is not death we have to contend with, but life.

Rien n'est la proie de la mort;  
Tout est la proie de la vie.

### ERRATA.

In Dr. Squire's paper on the processes concerned in the conversion of starch into alcohol and their relation to brewing and distilling, June 9th 1884, August number.

- Page 397, column 2—Date of meeting, for "May 5th," read "June 9th."  
 " 398, " 1—Line 21 from top, for "hang on to these electrical notions," read "hang on to electrical notions."  
 " 399, " 1—Line 3 from top, omit the words, "of these drawings the photographic reductions are exhibited on the adjoining page."  
 " 400, " 1—Line 4 from bottom, for "the influence the diastase," read "the influence of the diastase."  
 " 402, " 2—Last paragraph. The description of the wood cut should read thus:—

The experiment consisted in passing a parallel beam of light from the electric lamp through a large Nicol's prism A by which it was polarised (Fig. 3). The beam then traversed a trough B provided with plate glass ends *bb*, and a thin plate glass removable partition *c*, so as to divide the trough into two unequal cells. The light next encountered a plate of quartz C made up of two semicircles *d* fitted together, one of which consisted of dextro-rotary, and the other of levo-rotary quartz; both plates having a thickness of 3.75 mm. The light etc. etc.

Page 406, column 1—Line 10 from top, for "spent mash," read "spent wash."

### DISCUSSION.

MR. KINGZETT said that although he had not had the pleasure of hearing Mr. Squire's original paper, he had been much interested in the remarks made by

him that evening, and would like to make a few observations. As regarded the sterilising influence of an electric current, he would remind the meeting of the experiments made by Drs. Richardson and Wood in 1854, and of the investigations generally, conducted by Schönlein and Boillot, by which it was shown that ozone (generated by an electric current) exhibits very powerful disinfecting (and therefore sterilising) properties. Since then the progress of investigation had rendered it probable that these results were partly due to the peroxide of hydrogen, simultaneously produced with the ozone. Mr. Squire had attributed the discovery of the antiseptic properties of peroxide of hydrogen to M. Paul Bert and his collaborateurs, but as a matter of fact the credit was due to him (Mr. Kingzett). He had investigated the remarkable antiseptic properties of peroxide of hydrogen years since, and had communicated his earlier results to the Chemical Section of the British Association in 1876. Those results had since been amply confirmed, and in a letter he had recently received from M. Paul Bert, that gentleman had fully acknowledged his (Mr. Kingzett's) claim of priority, and had promised to make an announcement of it at an early meeting of the Academy of Sciences. Concerning the fermentation which took place upon the surface of the grapes, it struck him while listening to the remarks of Mr. Squire that possibly the bubbles of gas observed inside the grapes were not due to any active fermentation carried on within, but to diffusion from without, through the skin of the grape. He made this suggestion by way of a reasonable explanation of what Mr. Squire termed molecular action. He did not understand that expression in relation to micro-organisms, which were things that lived and moved and had their being: organisms which lived, so to speak, by eating and drinking, and which yielded excretory and secretory products. In connection with this subject, he would call attention to the valuable contribution recently given by Klein ("Practitioner," August, September and October, 1884), to the subject of the relations of micro-organisms to disease. He was glad to find that Klein, in seeking to explain the action of germ life as a factor in the production of disease, had come to the conclusion which he (Mr. Kingzett) had for many years advocated, viz., that the infectious diseases which result from the inoculation of the human body with germs, were not to be attributed to the mere presence of the microbes in the body, but to the necessary presence or production of definite chemical substances which act as specific poisons. Micro-organisms constituted an important order in creation, and one by whose agency dead organic matter, at large, was converted by hydration and oxidation into useful, innocuous, nay even, essential products. In practical disinfection, therefore, it was a mistake to direct measures to the extermination of micro-organisms; they should rather aim at controlling the life history of the germs, and the chemical destruction of the morbid products which constitute the real viri of disease.

MR. E. G. HOOPER said that commencing with the electric theory to which Dr. Squire had devoted considerable attention, he would agree with him as to the absurdity of the old-fashioned ideas of the effect of electricity on fermentation. It was perhaps as well that Dr. Squire had thoroughly cleared up the subject. One learned, however, to regard those old ideas as having in most cases some basis of truth, and in reflecting on this particular subject the fact was recalled that not only did the brewer suffer from the deterioration of his wort or beer, in what was termed thundery weather, but the house-wife, the butcher, and the poultryer also suffered at such times. To this fact he was inclined to attribute the



origin of the brewer's fear of electricity, although electricity was not to blame, but it was rather that in thundery weather the conditions were favourable to the development of putrefactive ferments. With regard to the action of malt at particular temperatures, Dr. Squire had expressed a difficulty in representing the action by an equation, and he thought they might well agree with him in that. They were dealing first of all with diastase, a body about which they knew very little; and with starch, a body more or less perfectly developed. If they took any particular sample the starch was found to consist of granules of varying size: and as these varied in size they might presume they varied also in other respects. He therefore thought that starting with an unknown quantity of active diastase and with starch in a varying state of development, they could never get an exact result to follow. Effort must be devoted to discovering more about diastase before they could go very far in the way of expressing by chemical equations its action on starch. He agreed with Dr. Squire's condemnation of the alleged osmotic action of diastase. There was no doubt that in the brewing operation what the brewer had to aim at was the production of such a temperature at should suffice satisfactorily to gelatinise the starch, and yet affect as slightly as possible the diastase. He believed it was quite impossible to perfectly gelatinise the starch without to a greater or less extent affecting the diastase, and at the same time he considered too much importance should not be attached to a table of gelatinising temperatures such as was given by Dr. Squire for as he had just contended, the gelatinising temperature was not invariable. Coming to the theory last developed by Dr. Squire, he (Mr. Hooper) said that one was taught in these days that all chemical action was ultimately to be referred to vibratory molecular motion, and it seemed like begging the question to speak either of bodies acting upon other bodies in a vibratory manner, or to speak of certain bodies as catalytic agents. That was simply to say they did not know what action those bodies exerted on other bodies with which they were brought in connection. The old theory was that catalytic bodies exerted some magic influence by their presence, but the later theory gave them a decided chemical action, if only a temporary one, and he thought that in those changes that were ascribed to vibratory motion, there was similar distinct chemical action, if, in point of time, it was limited. These like all other chemical changes might be based upon vibratory motion, but at the present time they could not say so; and until they were in a position to state exactly what did take place, he thought it was a very great pity that the question should be obscured, in reference to vibratory motion. With regard to Dr. Squire's grape experiment, he would remind the meeting of those conducted by Pasteur with respect to the production of alcohol in ripe fruits, severed from the tree and placed in an atmosphere of carbonic acid gas. If ripe fruits so immersed developed alcohol, he could not see any very great difference between Pasteur's experiment and Dr. Squire's. The next point he had to comment on was with regard to the acid produced during fermentation. Dr. Squire said that this acid was chiefly lactic acid, but he did not agree with him. Possibly there might be a large proportion of lactic acid in distillers' wash, but in brewers' wort which had been boiled, they would find that the acid which was mainly developed during fermentation was acetic acid. He would call attention to experiments made some years ago by the French chemist Duclaux, whose results confirmed by Béchamp, and admitted by Pasteur after considerable discussion, showed that in every case of alcoholic

fermentation acetic acid was produced. It was at that time proved that even in an atmosphere of carbonic acid gas, the amount of acetic acid developed amounted to 0.05 per cent. of the amount of sugar decomposed.

Mr. SALOMAN, referring to Dr. Squire's remarks concerning the sterilising action of the electric current, called attention to some experiments recently recorded in the Chemical Society's Journal, and made with peroxide of hydrogen, with a view to testing its efficacy as a preservative, when it was found to be absolutely useless. With respect to the remarks regarding Pasteur's experiments upon fruit, he thought, quoting from memory, that the whole gist of the experiment consisted in his having found that the germs had existed upon the walls of the fruit. Then, too, it was not at all necessary, while accepting Pasteur's theory, to contend that the alcohol was produced actually within the yeast cell. In all cases of life the particular action resulting from that life was manifested somewhere on the surface. For instance, the blood is oxygenated in the lungs, and the result is apparent on a surface in free contact with the general air, that did not differ to any considerable extent in any essential point from the yeast cell. Whatever might be the result of the yeast life of the plant, that result would be manifest somewhere on the surface of the yeast cell.

Mr. S. H. JOHNSON said that some few years ago he passed an electric current from an ordinary machine he was using through a solution of sugar washings, containing about 15 per cent. of cane sugar and three or four of grape. The cell was divided into three parts. The second part contained water acidulated to take the current, and the inner compartment contained the sugar solution. He found that the solution which had been acted upon would change after standing a week or so, while the original solution went sour in a few hours.

The CHAIRMAN said there was very much in what they had heard which was valuable. With regard to the question of molecular action he confessed that was a term which he viewed with aversion. It always seemed to him that the use of it was chiefly to conceal ignorance. The question was whether they could form any conception whatever of molecular action as distinct from chemical action, and whether they could conceive of molecular action apart from actual chemical action. The question after all was whether they were not introducing into the problem a factor of which they knew nothing, and the theory of which they were utterly unable to determine.

Dr. SQUIRE: I fear that the theory of molecular vibration does not meet with much favour this evening, but no one who is acquainted with molecular physics can fail to see how completely this theory harmonises with the views that have forced themselves on the greatest thinkers in all departments of natural science. It is now generally admitted that the molecules of all bodies are in a state of movement; in solid bodies the movements are vibratory only; in fluids they are vibratory and progressive; in gases they are progressive only. By heat these movements are intensified. Heat, then, is nothing more than the increased molecular movement of the body, which presents the phenomenon of being heated. That light is nothing more than molecular motion is unquestionably demonstrated. The vibrations have been counted, and their amplitude has been measured. Nay, more than that, they can be made evident to the eye by Fresnel's beautiful experiment of the inclined mirrors. When a ray of light is allowed to fall upon two mirrors touching one another, almost in the same plane, but very slightly inclined to one another, two rays of light are projected almost parallel, but slightly

converging. Under these circumstances the waves of light in the one ray cut the waves in the other, in such a way that the waves alternately intensify and neutralise one another, and thus produce a series of light and dark spaces visible to the eye. Here, then, we have an action of pure demonstrable molecular vibration. But a ray of light is capable of producing chemical decompositions, and not of one kind only. If we spread out the light by means of a prism, we obtain a spectrum. In the different coloured parts the vibrations are of different amplitude and of different frequency, and we find that one substance can be decomposed at one point of the spectrum, and only there, while another substance is decomposed at another point, and only there. Here we have a convincing proof that these decompositions are due to molecular vibration, and molecular vibration only, and that each decomposition requires a particular kind of molecular vibration. I confess I am quite unable to see in what other way the action of one ferment in restraining others can be explained. As long as the yeast cells are far apart there is no restraining action, but as soon as they approach within a distance, represented by the twentieth or thirtieth of a millimetre, no other ferment can grow in the intervening space. What is there in this intervening space which produces this effect? It is clearly not anything exuded from the yeast cell, for anything of this kind is favourable to bacterial growth. It is evidently some influence which the yeast cell is able to exert at a distance. Quite understand, I am not the inventor of this theory. I cannot claim to have done anything on molecular vibration myself. I am simply Nägeli's mouthpiece, as it were; but as far as my humble opinion has any weight, I endorse it thoroughly, and express my perfect conviction of its soundness. Take the case of the acetic acid ferment. Here we have an organism which cannot live without oxygen, which is greedy of that element. We should naturally expect that such an organism would exert a reducing rather than oxidising action. So it would, no doubt, if its action were confined to its own nutrition; if the chemical action were confined to the interior, or the actual surface, of the cell. But suppose that the life of the cell sets up an action at a distance, a molecular disturbance, by virtue of which the alcohol is oxidised in the presence of free oxygen, an action analogous to the disturbing influence of heat on the vapour of alcohol, by virtue of which it ultimately takes fire—then we can understand how an oxidising action may be set up in the neighbourhood of a cell, which *per se*—and if incapable of this action at a distance—would exercise a reducing action. Precisely the same argument applies to the mould organisms, which are still more greedy of oxygen, and yet exert such a powerfully oxidising action that organic substances are, through their agency, burnt to carbonic acid and water. As to electricity, it has been suggested that some foundation must have existed for the popular belief on this subject. No doubt that is so. When the weather is hot and close, things do not keep as well, and fermentations are more difficult to regulate, and it is just at this time that thunderstorms most frequently occur. Things that are equal to the same thing are equal to one another, but it does not follow that things which are caused by the same thing are caused by one another. Many popular beliefs have some foundation. Ninety-nine people out of a hundred will tell you that the sun puts the fire out, and so convinced are they of the fact, that I have quite given up arguing the point. No doubt it has this basis of fact. When the sun shines into a room everything looks bright, and warm, and cheerful; people forget all about the fire; they forget to poke it, and out it goes. It has been suggested by Mr.

Hooper that it is acetic rather than lactic acid, which is produced during the fermentation of beer, and, in support of this view, he urges that the worts have been boiled, by which the lactic ferment would be destroyed. He forgets, however, that this would also be the case with the acetic ferment. The acetic ferment is destroyed at this, and, indeed, at a much lower temperature, but, as a matter of fact, the lactic acid ferment is not destroyed at the boiling temperature if the worts are neutral or only very slightly acid. The lactic ferment stands out among all other ferments by its marvellous power of resisting high temperatures. No doubt in very deep coppers the temperature at the bottom of the vessel is much above the boiling point, and the lactic ferment might be destroyed under these circumstances, but unless great care and cleanliness be exercised it is very difficult to keep this ferment out of the worts afterwards. In distillers' worts the amount of lactic acid produced is stupendous, but that is mainly due to the high temperature at which the fermentation is conducted, and the absence of anything like hops to restrain that ferment, except the restraining influence of the yeast itself, and this action I have endeavoured to explain by the theory of molecular vibration. Still, I am not prepared to say that the whole of the acid in beer is lactic acid. Succinic acid is undoubtedly present, and acetic acid, if any be present, is easily detected by distillation, as the lactic acid is not volatile. It has been stated that peroxide of hydrogen has been tried as an antiseptic for beer, and has totally failed. I think that is very likely. I mentioned that, in the presence of yeast, permanent sterilisation by electrolysis could not be obtained owing to the decomposition of this substance by the yeast. Beer, in casks or in bottles, is never free from yeast corpuscles, so that we could hardly expect the peroxide of hydrogen to be of any practical value. This, however, does not prevent the peroxide of hydrogen from being an excellent antiseptic in the case of decoctions of hay, or similar substances, in which bacteria multiply with great rapidity, no yeast being present; whereas it would be of no use in the case of beer.

## Liverpool Section.

Chairman: E. K. Muspratt.

Vice-Chairman: Prof. J. Campbell Brown, D.Sc.

### Committee:

Ernest V. Bibby.  
Hudson A. Binney.  
Eustace Carey.  
John Hargreaves.  
E. Milner.  
E. W. Parnell.

C. Symes.  
G. I. J. Wells.  
F. Hurter, Ph.D.  
H. Brunner.  
A. Norman Tate.  
Dr. Affleck.

Local Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be sent to Edward Geo. Ballard, Queen's Park, St. Helens. There will be no meeting of this Section in December.

UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL,

Monday Evening, Nov. 5th, 1884.

MEETING.—A discussion on Instruction in Technical Chemistry was opened by Mr. A. Norman Tait, and was adjourned for further consideration to a future meeting of this section.



## Manchester Section.

Chairman: Sir H. E. Roscoe, LL.D., F.R.S.

Vice-Chairman: D. B. Hewitt, M.D.

### Committee:

R. F. Carpenter.  
C. Estcourt.  
H. Grimshaw.  
Peter Hart.  
J. von Hohenhausen.  
T. Jackson.

J. Knowles.  
I. Levinstein.  
C. Schorlemmer, F.R.S.  
Watson Smith.  
Wm. Thomson.  
D. Watson.

### Local Secretary:

J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

## Newcastle Section.

Chairman: J. C. Stevenson, M.P.

Vice-Chairman: B. S. Proctor.

Auditor: N. H. Martin.

### Committee:

P. P. Redson, D.Sc.  
G. T. France.  
C. H. Gimingham.  
John Glover.  
A. S. Herschel, M.A.  
John Morrison.

John Pattinson.  
H. R. Proctor.  
W. W. Proctor.  
J. F. Stark.  
T. W. Stuart.

Local Secretary and Treasurer: J. T. Dunn, M.Sc., 115, Seotswood Road, Newcastle.

MEETINGS, SESSION 1884-5, Dec. 4th, 1884; Jan. 8th, Feb. 5th, March 5th, 1885.

December 4.—Discussion on Dr. Divers Paper.

" Discussion on "Relative Advantages of Machinery and Hand Labour," to be opened by the Chairman.

Meeting, Thursday, November 6, in the Lecture Theatre of the College of Science.

MR. J. C. STEVENSON, M.P., IN THE CHAIR.

## ON THE CHANGES IN ALKALI-WASTE WHICH GOVERN THE RECOVERY OF ITS SULPHUR.

BY EDWARD DIVERS, M.D., PRINCIPAL OF THE IMPERIAL JAPANESE COLLEGE OF ENGINEERING, TOKIO.

ALTHOUGH the manufacture is languishing in which alkali-waste is produced, and some chemists are turning away in despair from the Leblanc soda-process, the utilisation of the waste remains of importance and is still being made the subject of fresh patents. A successful attempt by T. Shimidzu, M.E., and myself to prepare crystals of pure calcium hydrosulphide, described in the July number of the *Journal of the Chemical Society*, led us to observe and investigate some points in the action of water and air upon calcium sulphide, which seemed to require elucidation.

Our results are so closely related to the problem of the advantageous extraction of sulphur from alkali-waste, that I believe an account of them in this relation may prove worthy the attention of those interested in the matter and of others. I will place my remarks under two headings, one being hydration, including hydrolysis, and the other oxidation.

*Hydration.*—During the period in which soda-waste is left in heaps to ripen in the air, the calcium sulphide, of which it mainly consists, undergoes hydration as the preliminary to all other changes. This is a statement of what now appears to be generally accepted as a fact. Kraushaar first called

particular attention to it in connection with soda-waste utilisation. Dry calcium sulphide will indeed oxidise when heated, but, even then, only to sulphite and sulphate, not to thiosulphate, or polysulphide; and it is not decomposed by carbon dioxide. For a long time in the history of attempts at sulphur-recovery, oxidation was held to be the essential change preliminary to lixiviation, but the various patents taken out since Kraushaar's experiments, show that hydration is now commonly recognised as the necessary change; and where the idea once used to be to hasten oxidation, it is now rather to hasten the resolution of the sulphide by water without oxidation.

Did calcium sulphide readily take up water and dissolve, or yield a soluble sulphur compound, much of the difficulty of sulphur recovery would disappear, but this it does not do. Why this is the case it is now possible to explain to some extent; and also why, since hydration must precede other changes, oxidising and carbonating should appear to be much more rapid in breaking down the sulphide than the very process preceding them. But to do so, I must first give some description of the newly discovered calcium hydrosulphide, and of the undoubtedly commonly occurring, but hitherto almost as little known, calcium hydroxyhydrosulphide.

*Calcium hydrosulphide.*—Is a colourless, crystalline salt which can only exist at common temperatures and in an atmosphere of hydrogen sulphide. This instability is seen in the crystals, which contain six molecules of water, rapidly deliquescent when exposed to the air into a mass of wet crystalline particles—not, however, by the absorption of any water from the air, but by the loss of hydrogen sulphide—the wet residue consisting of some of the water of crystallisation and of calcium hydroxyhydrosulphide. The decomposition of a concentrated solution of the hydrosulphide is no less remarkable than that of the crystals; for this, too, when exposed to the air evolves great quantities of hydrogen sulphide and becomes quickly coated over with a crystalline crust of pure hydroxyhydrosulphide; or, when air is blown through it, soon becomes filled with a crystalline precipitate of the same salt. This instability is quite independent of any action of either oxygen or carbonic acid; and, remarkable to state, with neither mode of exposure of the solution is there any absorption whatever of carbonic acid, or any more than the slightest absorption of oxygen. The hydrosulphide is exceedingly soluble, the crystals requiring no more than a fourth of their weight of water to dissolve them, and the solutions reach a specific gravity between 1.30 and 1.35. It was not prepared from calcium sulphide in such concentrated solutions as these and in crystals, but by bringing together quicklime and sulphuretted hydrogen in a sufficiently limited quantity of water, and crystallising the salt by cooling. In order to succeed, much time is required to dissolve the lime, and not more than about two and a half parts of water must have been added to one part of pure quicklime. Calcium sulphide suspended in water also forms the hydrosulphide when treated with sulphuretted hydrogen, but only slowly and so as to suggest the previous hydration of the sulphide.

*Calcium hydroxyhydrosulphide.*— $\text{CaSH.OH.3Aq.}$ , would by many chemists be described as "hydrated calcium sulphide,"  $\text{CaS.4Aq.}$ , a difference discussed in our paper in the *Chemical Society's Journal*. This difference is, however, not one without significance to those occupied in tracing out the nature of the formation of this salt from the anhydrous sulphide and of its decompositions by air and water. For the latter title represents no chemical change to have taken place by the hydration in the position of the sulphur

to the calcium, shutting our eyes to what is surely the fact, that not merely the hydration but the hydrolysis of the sulphide has proceeded a stage; and, unlike the former, does not throw any light upon the way in which the sulphide may pass by hydration and oxidation into thiosulphate and pentasulphide.

The existence of this salt in the form of minute acicular crystals was observed by Berzelius; but crystals of it of some size, four-sided prisms, can also be prepared, though with difficulty. It is colourless and moderately soluble in water, but when in solution it almost at once separates into calcium hydroxide insoluble, and the very soluble hydrosulphide. In calcium hydrosulphide solution of any great degree of strength it is insoluble, and is not decomposed by the water of such a solution. Indeed, when a solution of lime in sugar water is added to a solution of calcium hydrosulphide, there is an instant and copious precipitation of the hydroxyhydrosulphide; and solid lime, agitated in a state of fine division, with a concentrated solution of calcium hydrosulphide, unites with this to form the hydroxyhydrosulphide then insoluble. It loses hydrogen sulphide on exposure to air, and absorbs both carbonic acid and oxygen, but, as I shall afterwards explain, the absorption of the latter is apparently indirect. By heat both the hydrosulphides are decomposed, and in a current of dry hydrogen sulphide the residue is principally calcium sulphide, otherwise it is for the most part calcium hydroxide. Besides being formed in the two ways described, it is also formed by the union of sulphuretted hydrogen and slaked lime, and doubtless by the hydration of calcium sulphide. Beyond doubt too, the sulphuretted lime of the coal gas "purifiers" must contain much of this body. Some experiments by Folkhard, published in the *Chemical News* just after our paper was read at the Chemical Society, in which dry hydrogen sulphide mixed with some hydrogen was passed for some hours over slaked lime gave a product having the composition  $4\text{CaO} \cdot \text{H}_2\text{S} \cdot 3\text{SH}_2$  but with no claim to be regarded as a definite compound. Had Folkhard used pure but slightly moist sulphuretted hydrogen he would no doubt have got unmixed hydroxyhydrosulphide.

Having thus prepared the way, I may now offer an account and explanation of the behaviour of alkali-waste in course of hydration and decomposition, although not improbably much of what I am going to state will have been gleaned from my description of the hydrosulphides.

The calcium sulphide of the waste certainly becomes in the first place calcium hydroxyhydrosulphide, and is the source of the hydrosulphide found in the lixiviation waters. The experiments of Kolb and others have indeed shown how very slowly calcium sulphide yields any hydrosulphide to water, but Kraushaar's experiments prove that without any material oxidation, the sulphide does undergo considerable hydration. Of this slowness in hydrating I have no explanation to offer, nor of the remarkable and important fact, for I believe it is one that the process of hydration goes on to only a limited extent until the mass has been lixiviated. But as to the relatively rapid manner in which further solubility of the sulphur as pentasulphide and thiosulphate is gained by blowing air through the moist mass, that is evidently entirely due to the hydration being favoured by the heat arising from the oxidation going on. Any hope of lixiviating hydrated waste so as to get concentrated liquors must be given up in consequence of the hydroxyhydrosulphide being both insoluble in calcium hydrosulphide solution and undecomposable by it into lime and the hydrosulphide. Whether the hydroxyhydrosulphide is less stable and insoluble in oxidised or "yellow" liquor, as it would have to be in

order to make Simpson's recently patented process a success, I do not know. Practised with exposure to the air, the use of "yellow" liquor is certainly unworkable as Mactear has shown, in consequence of the large evolution of hydrogen sulphide which takes place. Of this peculiar evolution I shall offer some explanation near the end of this paper.

The attempts to get nearly all the sulphur of the waste quickly into solution as calcium hydrosulphide by heating the waste with water under pressure, have been attended with two difficulties—the large proportion of water required, and the large quantity of hydrogen sulphide set free. The former of these is due to the fact just noticed in treating of cold lixiviation, that although calcium hydroxyhydrosulphide, the hydrated calcium sulphide, dissolves in simple water, and then decomposes into lime and calcium hydrosulphide, it is yet insoluble in a solution of calcium hydrosulphide of much strength; and this difficulty is probably insuperable. The other difficulty is in consequence, of course, of the ready convertibility of hydrosulphide solution, when concentrated, into hydroxyhydrosulphide and sulphuretted hydrogen, and, when dilute, into lime and this gas. When a moderately strong solution of the hydrosulphide is boiled in an open vessel, calcium hydroxide precipitates; and a current of air through water in which the hydroxyhydrosulphide is suspended, slowly converts the latter into calcium hydroxide.\* In agreement with this statement, Pechiney has found that on blowing air and steam through yellow liquor much calcium hydroxide gets precipitated (Lunge). Even in the cold, the weakest solution smells of hydrogen sulphide, the liberation of this gas being independent, as I have already pointed out, of any chemical action of the air. I cannot think that any means will be found that will allow of a concentrated solution of calcium hydrosulphide being prepared by the hydration of alkali waste.

Opl's earlier process of decomposing alkali waste by carbon dioxide in presence of water, and passing the liberated hydrogen sulphide into a second quantity of wet waste, so as to convert the sulphide of this into hydrosulphide, must have certainly failed, for several reasons. One, already pointed out by Weldon, is the impossibility of keeping carbon dioxide out of the second quantity. This gas is not nearly so completely absorbed by a dilute solution of hydrosulphide as may be supposed; indeed, hydrogen sulphide can dissolve any quantity of precipitated calcium carbonate if a sufficiently rapid stream of it be kept up. Another difficulty is that the available carbon dioxide is largely diluted with nitrogen and other gases, so that even if all of it could be retained in the first vessel of waste, these other gases would carry off from the second vessel large quantities of hydrogen sulphide unabsorbed, because of the fact that calcium hydrosulphide is not permanent in an atmosphere of any gas but hydrogen sulphide. A third difficulty is that calcium sulphide is too slow for technical purposes in combining with hydrogen sulphide, according to our experiments made on a small scale. A fourth difficulty is that the calcium hydroxide, and some, at least, of the carbonate, always present in the waste, have also to be saturated with hydrogen sulphide in the second vessel.

Opl's later process, or rather, Opl and von Miller's process, is equally unpromising. A solution of calcium hydrosulphide having been prepared from alkali waste, this is to be made, by boiling, to give up its

\* Partly also into tetrasulphide by oxidation, which, combined with the lime, constitutes the Rose-Schöne salt.



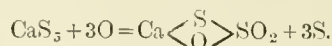
hydrogen sulphide. Part of this is to be converted to sulphur or sulphuric acid at pleasure, and the rest is to be used in preparing a fresh solution of calcium hydrosulphide by dissolving out with it in water the sulphide of alkali waste. Not only are there the objections which apply to the other process, that hydrogen sulphide by no means quickly and readily dissolves calcium sulphide, and that it has to saturate the free lime in the waste, but there are obviously others. A solution of calcium hydrosulphide only slowly decomposes, even when vigorously boiled, unless the atmosphere over it is rapidly changed and the liberated hydrogen sulphide in this way carried off; and, therefore, the expulsion of the last portions of hydrogen sulphide will involve the generation of much steam, and this steam has to be condensed and the gas cooled before it comes to the moist waste. Another difficulty to be met, is to force this gas into the closed vessel holding the waste, since without pressure it could never be passed with any speed into the waste and yet be all absorbed.

There seems to me one way of working alkali waste for hydrogen sulphide, which is worthy the trial and consideration of those interested. This is to force steam through a series of about four closed vessels of waste, successively charged and emptied, as in the working of a set of lixiviation tanks. Sulphuretted hydrogen will be found to escape from the last vessel sufficiently fast for utilisation, I believe. Were there a cheap enough source of carbon dioxide unmixed with any other gas, this, too, might be used to assist the steam, but that source is not yet discovered, nor likely to be.

*Oxidation.*—By spontaneous and forced oxidation of moist alkali waste, alternated with lixiviation, a mixture of calcium polysulphide and calcium thiosulphate is obtained, or ultimately thiosulphate alone. The polysulphide used to be considered to be the bisulphide; it is now known to be both the tetra- and penta-sulphides, but for simplicity I may here be allowed to treat of the pentasulphide only.

The facilities which chemical equations afford the chemist in enquiring into and explaining chemical reactions have often been abused, but probably in no case more than in the consideration of the action of the air in forming thiosulphate and pentasulphide from the simple sulphide. Equations have been put forward, not only without any basis of facts—other than those to be interpreted—but even in direct opposition to facts. Thiosulphate contains two atoms of sulphur to one of calcium, therefore the polysulphide of “yellow” liquor has been conveniently assumed to be bisulphide. Calcium sulphide, two molecules, has been represented to oxidise into oxide and bisulphide, although no one can perform the feat of replacing by free oxygen sulphur that is combined with calcium. Oxidation of calcium sulphide does not occur in the cold, and at high temperatures gives sulphite and sulphate, but neither polysulphide nor thiosulphate, as it is made out to do. Having thus got bisulphide, the next step has been to represent this as combining with three atoms of oxygen to form the thiosulphate. Now, no fact is better established in chemistry than that we have in calcium thiosulphate the metal half combined with sulphur and half with oxygen. How, then, are we to understand this conversion

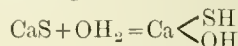
phide changes into thiosulphate and sulphur, is the following—



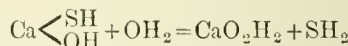
and is just as difficult to understand and to admit as the first one. But here I hardly presume to complain of its acceptance, since facts have seemed to be so strongly in its favour. Indeed, Mr. Shimidzu and I have by careful experiments convinced ourselves that, starting with pure calcium pentasulphide in solution and exposing this to air free from carbon dioxide, we get the whole of the calcium as thiosulphate simply, and three-fifths of the sulphur in the free state. But then I must add that other experiments have proved to us that this change of pentasulphide can be resolved into stages; and in these successive changes the three atoms of oxygen do not displace any sulphur at all.

In the consideration which follows of what really appears to happen in the oxidation of alkali waste, I have made few assumptions, but have based my view of the matter for the most part upon ascertained facts. But since what I have to advance is much opposed to current notions on the subject, it runs some risk of being set down in haste as too theoretical.

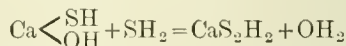
I have already insisted upon the point that all other changes in alkali waste are preceded by that of hydration, and it is evident that this hydration is a process of *hydrolysis*, or chemical decomposition by water, the stages being—



and—



besides the secondary reaction—



When the moist waste lies in a covered-in heap there will be present, therefore, besides unchanged calcium sulphide, calcium hydroxyhydrosulphide, calcium hydrosulphide, calcium hydroxide, and hydrogen sulphide. It may shock our old-fashioned notions about acids and bases, but modern work, especially in thermo-chemistry, seems to leave it beyond doubt that weak acids and acid-salts may, in dilute solution, be indifferent to free bases present in the same solution. Moreover, the fact that inactive gases such as nitrogen and hydrogen, blown through moist calcium sulphide will carry off hydrogen sulphide continuously, is conclusive as to the correctness of the above statement. It is quite true that some of these will be present in only minute proportions at a time, but then it is equally true that we have only to remove these quantities by some means to have them replaced at once by fresh ones.

The atmospheric oxygen comes, then, nowhere in contact with the components of moist alkali-waste that it does not find hydrogen sulphide among them in minute but practically irreducible quantity—thanks to its hydrolytic generation as fast as oxidised; and the supply of air can be so regulated that no hydrogen sulphide escapes from the mass. Now, we already know for certain that moist hydrogen sulphide oxidises readily but slowly; although I know of no experiments having been made to test its rate of oxidation under the most favourable circumstances. But some simple experiments made by Mr. Shimidzu and myself, which gave very remarkable results, are to the point in this connection. Rapid currents of decarbonated air were sent for six hours through



of bisulphide into thiosulphate by oxygen? Another common equation which serves to represent how, by the further oxidation of “yellow” liquor, pentasu-

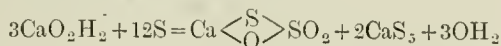
solutions of calcium hydrosulphide and of pentasulphide, and through water having calcium hydroxy-hydrosulphide in excess suspended in it: in all cases, *but mere traces of thiosulphate were produced*, and in the hydrosulphides, *but mere traces of pentasulphide*, while *hydrogen sulphide was carried off continuously* from all. In the residues, besides undecomposed salt, *much calcium hydroxide*, free or combined with tetrasulphide, was found, and in the residue of the pentasulphide, *much free sulphur*. These results show that the direct oxidation of the hydrosulphides must be quite as slow a process as that of hydrogen sulphide, if, that is to say, it occurs at all; for as to the minute quantity of oxidation products obtained by us, this could well have come all from the hydrogen sulphide alone. Whether the calcium hydrosulphides can oxidise or not, does not admit of being tried, so far as I can see, because they continue to yield hydrogen sulphide as fast as it is removed, and this appears sufficient to consume all the oxygen absorbed; for the oxidation of the waste is itself a very slow process in the cold, and as regards the greater rapidity of action of air forced in, the heat then accumulating in the mass would cause this to be exerted as much upon hydrogen sulphide as upon any of the other components of the waste. The conclusion thus forces itself upon us, that *in moist waste*, or any other form of calcium sulphide and hydrosulphides, *it is hydrogen sulphide which oxidises, and that the calcium salts do not oxidise at all*.

To admit that hydrogen sulphide by oxidising may contribute, to a limited extent, some of the sulphur of the persulphide of the "yellow" liquors is no novelty;\* but this is a very different matter from that of adopting the view that I have here enunciated, that *the entire oxidation of alkali-waste is confined to that of hydrogen sulphide set free in it*.

The next point to determine is to what extent the hydrogen sulphide becomes oxidised—whether, that is, its hydrogen alone oxidises, or whether both hydrogen and sulphur oxidise. It is difficult to emancipate ourselves from the rule of long-accepted doctrines, but if we independently examine into the view that the production of the calcium thiosulphate of oxidised alkali-waste is due to the oxidation of the sulphur—whether free or combined does not matter—by the atmosphere, we shall see that it is hardly justified by the facts, as being on the one hand unnecessary, and as affording, on the other, no simple explanation of that production. Unoxidised sulphur is needed in any case, to account for the production of the pentasulphide, and the best equation I can construct to represent the formation of the thiosulphate by oxidation is far from being satisfactory. It is not to be denied that sulphur and hydrogen sulphide can and do oxidise in the air to acids, but the rate at which they do so seems as undeniably too slow to account for the production of thiosulphate in alkali-waste. It is also true that sulphurous acid forms thiosulphate with calcium hydrosulphide, but then only because of the reduction of some of the

sulphurous acid by hydrogen sulphide, and the combination of the sulphur thus formed with calcium sulphite. Such successive oxidation and deoxidation of sulphur in alkali-waste must be regarded as unlikely to occur, and we have therefore to adopt the view that if sulphurous acid acts, it is aided in its action upon lime by sulphur resulting from atmospheric oxidation. But if lime and sulphur alone should seem to be sufficient to furnish not only the thiosulphate but the pentasulphide also, we need not look for help from sulphurous acid, or for its formation at all. It is sufficient and less hypothetical to consider that *the atmospheric oxidation of alkali-waste is limited to that of the hydrogen of hydrogen sulphide generated in it*. This I shall proceed to show by giving an account of the formation of the pentasulphide and thiosulphate in alkali-waste, without assuming further oxidation than that required to furnish sulphur.

It is a well-known fact that sulphur and lime boiled together in water yield calcium pentasulphide and thiosulphate, the reaction of the two being expressed by the equation—



but this reaction seems not to have suggested itself to any one, as occurring in alkali-waste. Yet to allow this to be the case requires only the simple and reasonable assumption that, in the cold, sulphur liberated in intimate contact with calcium hydroxide—nascent sulphur, is as active as ordinary sulphur at a boiling temperature. It is no mere supposition that sulphur may act upon calcium hydroxide in preference to hydroxyhydrosulphide or hydrosulphide. For although sulphur can indeed act upon calcium hydrosulphide, changing it to pentasulphide and hydrogen sulphide, still it is necessary that the solution of the hydrosulphide should be concentrated; since if it is very dilute the action is only very slight. To this is to be added that hydrogen sulphide, on the other hand, readily decomposes calcium pentasulphide, forming calcium hydrosulphide and precipitated sulphur. It is therefore only in accordance with facts to hold that sulphur may act preferentially upon the calcium hydroxide present with the hydrosulphide.

The chemistry of the decomposition of alkali-waste by air and water, becomes thus reduced to a state of great simplicity: the ultimate products of the hydrolysis of calcium sulphide are calcium hydroxide and hydrogen sulphide; the latter of these is oxidised by air into water and sulphur; and the sulphur and the calcium hydroxide react to form calcium pentasulphide and thiosulphate, according to a well-known chemical reaction. Certainly, in hot moist waste during forced oxidation, this reaction must occur, even though it may not be the only one.

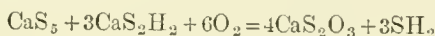
The result of the changes proceeding as here indicated would be the possibility of having in a "yellow" liquor much pentasulphide; never, from the very first, an absence of thiosulphate; and, if pentasulphide were itself permanent, for every one part of sulphur present as thiosulphate, five parts present as pentasulphide, as shown by the equation already given. Now, in accordance with this, it does prove impossible to get calcium hydrosulphide or alkali-waste to oxidise without finding both salts present from the first; and, secondly, Kraushaar has found as much as 40 per cent. of the sulphur as polysulphide, and in several samples of the earlier "yellow" liquors more than two parts sulphur as pentasulphide to one as thiosulphate, which, considering that the pentasulphide must be simultaneously changing into thiosulphate, is as high a ratio as we need expect to find,

\* The liberation of hydrogen sulphide recognised as needing the above admission, has been attributed to the action of carbon dioxide upon the waste, and on this point I should like to say a word. The carbon dioxide of the atmosphere has, I believe, no appreciable power of decomposing alkali-waste in its earlier states. I believe this, not merely because the waste contains a good deal of calcium hydroxide, and is constantly getting more of it through the hydrolysis of the sulphide, but because also of the fact that, though carbon dioxide undiluted freely decomposes calcium hydrosulphides, sulphuretted hydrogen freely also decomposes calcium carbonate, so that it is even possible to run a stream of the mixed gases through lime-water without causing any precipitation of carbonate. There is, of course, no doubt as to old or exhausted waste carbonating in the air, or as to the effect of blowing chimney-gases through waste.

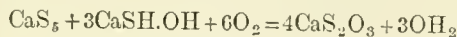


As for the after-conversion of the pentasulphide itself into thiosulphate, we have to recognise, first, the occurrence of its hydrolysis into calcium hydroxide, hydrogen sulphide, and sulphur, a fact already shown in this paper to be established; secondly, the atmospheric oxidation of the hydrogen of the hydrogen sulphide; thirdly, the reaction between the sulphur and this and additional lime to form thiosulphate, with, again, some pentasulphide; and, fourthly, a succession of these changes by which the pentasulphide recurs in less and less quantities down to nothing.

The after-conversion of the pentasulphide into thiosulphate can take place in this manner completely only when it is in contact with the lime of the waste. Where it is decomposed by the air in absence of lime, but two-fifths of its sulphur can become thiosulphate, the rest being left free; while if the contact with air is too extensive, hydrogen sulphide will escape and some lime be left, with, of course, less even than two-fifths of the sulphur as thiosulphate. Lastly, when "yellow" liquor is used to lixiviate fresh waste, the supply of lime will be too small for the conversion of all sulphur into thiosulphate when the lixivium reacts with the air, and there will consequently be sulphur deposited and hydrogen sulphide will escape. It was escape of hydrogen sulphide from this cause that rendered this mode of lixiviating unmanageable in the hands of Mactear. Omitting intermediate stages, the equation giving the result will show this—



and with a greater proportion of pentasulphide, or by the action of still more oxygen, sulphur will be deposited. A thick paste of "yellow" liquor and solid waste may (theoretically) be oxidised by the air, without liberation of either sulphur or hydrogen sulphide, thus—



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The next meeting will be held on Dec. 2, when Mr. W. J. A. Donald will read a paper on "The Chrome Tanning Process."

*A Meeting was held in the Rooms, 207, Bath Street, on Tuesday, May 13, 1884.*

— MR. E. C. C. STANFORD IN THE CHAIR.

## ON THE COMPOSITION OF TOBACCO.

BY JOHN CLARK, PH.D., ETC.

THE tobacco plant is very extensively cultivated in various parts of the world, and after it has reached its maturity it is cut and dried on poles. When the plant is in proper condition, the leaves are stripped from the stalk, sorted and cured, by which means they are converted into the tobacco of commerce. The good leaves are called "wrappers," and the infirm or defective ones, which are separated from the others, are called "mediums and fillers." The term "strips" is applied to tobacco leaves, from which 20 to 25 per cent. of the stem or midrib has been removed to suit the requirements of manufacturers in this country more especially. Tobacco is largely imported into the United Kingdom, partly in the manufactured state, but principally in the unmanufactured or leaf form.

Through the kindness of a well-known firm of tobacco manufacturers, I have been furnished with authentic samples of the principal varieties of leaf tobacco, imported into this country, and the accompanying table gives the proportions of mineral matter or ash, alkaline salts, and sand, which these contain. For the sake of comparison the results are all stated in the dry tobacco, and in order to ensure greater accuracy, the analysis was, in each case, made with several leaves, which were separated into laminae and stem, and the whole of each incinerated. The difference in the composition of the laminae and the

stem is very marked, especially as regards alkaline salts, and is of importance more especially to the snuff manufacturer.

rule from 14 to 22 per cent. Cigars, even the cheapest, are comparatively dry, and contain, as a rule, only from 10 to 12 per cent. of water.

COMPOSITION OF VARIOUS KINDS OF LEAF TOBACCO.									
WHOLE LEAF. Dried at 212° F. per cent.			LAMINÆ. Dried at 212° F. per cent.			STEM. Dried at 212° F. per cent.			
	Ash.	Alk. Salt.	Sand.	Ash.	Alk. Salt.	Sand.	Ash.	Alk. Salt.	Sand.
U. S. Kentucky .....	19.11	6.81	2.57	18.93	5.43	3.06	21.69	13.51	.68
do. ....	18.50	6.68	1.82	15.50	2.77	2.39	26.07	16.68	.38
do. ....	25.99	9.69	3.51	24.88	6.70	4.17	29.36	20.01	1.10
do. Strips.....	15.73	4.31	2.61	15.57	4.07	2.71	16.95	6.35	1.37
U. S. Missouri.....	20.96	5.07	4.63	20.46	2.62	5.27	22.61	12.72	1.90
do. ....	22.01	6.32	3.51	21.36	4.96	3.88	23.62	12.37	1.53
do. ....	18.88	4.81	2.61	17.18	2.88	3.21	22.17	10.68	.92
do. ....	18.36	4.60	3.44	17.05	2.50	4.07	22.39	11.10	1.49
U. S. N. Carolina .....	11.50	5.99	.63	12.98	3.92	.74	18.61	11.72	.23
Paraguay .....	30.80	8.15	12.32	31.07	6.37	14.41	30.37	14.78	4.91
Brazil—Carmen .....	20.51	7.81	.42	20.42	7.24	.46	26.86	9.37	.31
Holland .....	21.83	11.37	.13	20.16	8.99	.55	25.15	17.20	.12
Turkey—Cavallo .....	13.79	5.05	3.06	12.47	2.91	3.45	18.11	11.76	1.87
do. Latakia .....	19.50	7.19	.55	21.86	8.28	.72	15.41	7.73	.21
do. Samsoun.....	18.39	6.98	.49	17.59	5.32	.41	21.72	13.42	.60
Japan .....	15.67	6.86	.50	14.60	5.59	.54	19.81	11.55	.35
China .....	18.58	2.40	6.30	17.91	1.66	6.94	20.57	5.27	3.61
Havana .....	20.99	8.19	1.02	20.91	7.51	1.01	21.02	10.33	.92
Manilla .....	21.80	6.54	.14	21.25	5.49	.13	22.50	9.09	.11
German .....	22.27	3.76	1.79	22.12	2.78	1.87	23.13	4.63	1.39
Sumatra .....	18.61	7.20	.13	18.71	6.59	.09	18.11	9.11	.28

	Average of Whole Leaf per cent.	Average of Laminæ per cent.	Average of Stem per cent.
Ash or Inorganic .....	20.32	19.21	21.92
Alk. Salts .....	6.47	4.98	11.41
Sand .....	2.48	2.86	1.15

The unmanufactured tobacco which is imported into this country, is converted into roll or spun tobacco, cut tobacco and cigars, and the refuse is used for making snuff. Roll tobacco is the staple manufacture in Scotland and Ireland, and cut tobacco the staple article in England.

In the manufacture of roll tobacco, the leaves are moistened with water, spun into various sizes of twist, made up into rolls, and pressed. The liquid or juice which exudes under pressure is used as a sheep dip. Cut tobacco is made by moistening the leaves, cutting them into the desired size, and drying on plates. Sometimes it is made into cakes in the first instance, and afterwards cut.

When we compare the composition of roll and cut tobaccos with that of the leaf from which they are made, we find that the difference lies almost entirely in the amount of moisture, and as manufacturers are not allowed to add anything but water and a little oil to tobacco, you will not err very much in assuming that as a rule the cheapest qualities of roll and cut tobaccos contain most water. Thus in 15 samples of the cheapest roll tobacco I found an average of 41.66 per cent. of water.

The lowest qualities of cut tobacco, such as are largely manufactured and consumed in England, contain as much water as the cheapest roll tobacco, whereas the finer qualities of cut tobacco contain as a

The difference in cheap cigars is due chiefly to the weight of the material, but also to the quality of the tobacco and the labour, machinery being used in the manufacture of the lower qualities, whereas the higher qualities are nearly all hand made.

The large quantity of water contained in the cheapest tobacco, and which frequently amounts to about 50 per cent., is not, in my opinion, introduced to please the palate of the working man, but simply on account of the keen competition between rival manufacturers, and the low price at which tobacco is sold; and in the interest both of the working classes and of tobacco manufacturers themselves, I think it is very desirable that some limit should be placed to the amount of water which may be sold as tobacco.

Snuff.—I stated that the refuse tobacco was employed in the manufacture of snuff. This refuse consists of stems, tobacco smalls, and sweepings. These are moistened with water, subjected to a process of fermentation, which lasts from about six weeks to two months, then ground, mixed with alkaline salts to preserve the snuff, and flavoured when desired. Nothing is allowed to be added to snuff except the carbonates, chlorides, and sulphates of potash and soda, and carbonate of ammonia. It is also provided by Act of Parliament that any snuff found to contain, after being dried at 212° F., more than 26 per cent. of such salts, including those naturally in the tobacco, will be liable to forfeiture and a penalty of £50. From my table of analyses you will observe that not only does the proportion of alkaline salts vary in different tobaccos, but the stem contains a much larger proportion than the



leaf. On this account it is necessary that the snuff manufacturer should know the quantity of alkaline salts in his snuff material, in order to obtain an article of uniform composition. Some manufacturers go by rule of thumb, and in attempting to work close to the legal limit, they run a serious risk of unintentionally incurring the penalty. As a matter of fact, three samples of snuff, in 1883, were condemned by the Somerset House Authorities because they contained an excessive proportion of alkaline salts, and the manufacturers were prosecuted. The more intelligent of the snuff manufacturers, however, analyse their snuff material, and are thus able to keep within the legal limit.

The principal alkaline salts which are added to snuff are chloride of sodium or common salt, carbonate of potash, and carbonate of ammonia, all of which are allowed by Act of Parliament, and therefore no exception can be taken to their addition, so long as the total quantity does not exceed 26 per cent. in the dry snuff. In addition to alkaline salts snuffs usually contain from 25 to 45 per cent. of water, with the exception of a kind of snuff called "High Toast or Irish Blackguard," which is very dry and contains from 5 to 8 per cent. Sometimes they also contain a considerable quantity of sand. In the several hundred samples of snuff which I have had occasion to examine for different manufacturers the average quantity of sand was about 5 per cent. in the dry snuff, and sometimes fell as low as a half per cent., but in many samples the quantity exceeded 10 per cent., and in one case I found as much as 30.94 per cent. of sand in the dry snuff. The greater part of this sand is probably derived from the sweepings of tobacco, on which duty has been paid, and I have no doubt the snuff manufacturer considers himself justified in selling it as snuff. But it appears to me to be very desirable in the interest of snufflers, that some limit should be placed on the quantity of sand which may be sold as snuff: more especially as the particles of sand are frequently very sharp, and have a tendency to produce inflammation of the mucous membrane of the nose, and it is to this, probably, that we owe the popular notion that snuff is sometimes mixed with ground glass to give it additional piquancy.

When from any cause snuff is spoiled, the manufacturer may export it, and obtain a drawback of 3s. 7d. per lb. on the real tobacco which it contains.

The Government standard for tobacco is as follows:

	Per cent.
Organic Matter.....	70.52
Inorganic.....	15.48
Water.....	14.00
	<hr/> 100.00

This is equal to 18 per cent. of ash or inorganic matter in the dry tobacco. This standard is in my opinion too high as the average percentage of inorganic or ash in the dry leaf tobaccos which I have examined is 20.32, and the stem from which snuff is largely made contains still more. The result is that the tobacco manufacturer not only loses the value of the tobacco over and above the duty, but also a part of the duty which he has paid. This matter concerns the tobacco manufacturer alone, but I would point out that the authorities in Somerset House in fixing such a high standard for tobacco are benefiting the public at the expense of the manufacturer, whereas in the case of milk the low standard which they employ is a loss to the public and gain to the dishonest dealer.

#### DISCUSSION.

Dr. CLARK, in reply to a question of the Chairman, said that he did not discuss the question of the com-

position of the ash, or of the alkaline salts of the of the tobacco plant, as that had been very well investigated. The alkaline salts consist chiefly of carbonate of potash, chloride of potassium, and sulphate of potash.

### Obituary.

GEORGE WILLIAM WIGNER, F.I.C.,  
F.C.S.

*President of the Society of Public Analysts.*

Died October 17th, 1884, aged 42 years.

GEORGE HENRY HERBERT,

*Of the Firm of C. O. McAlum and Herbert,  
of Newcastle-on-Tyne.*

Died November 9th, 1884.

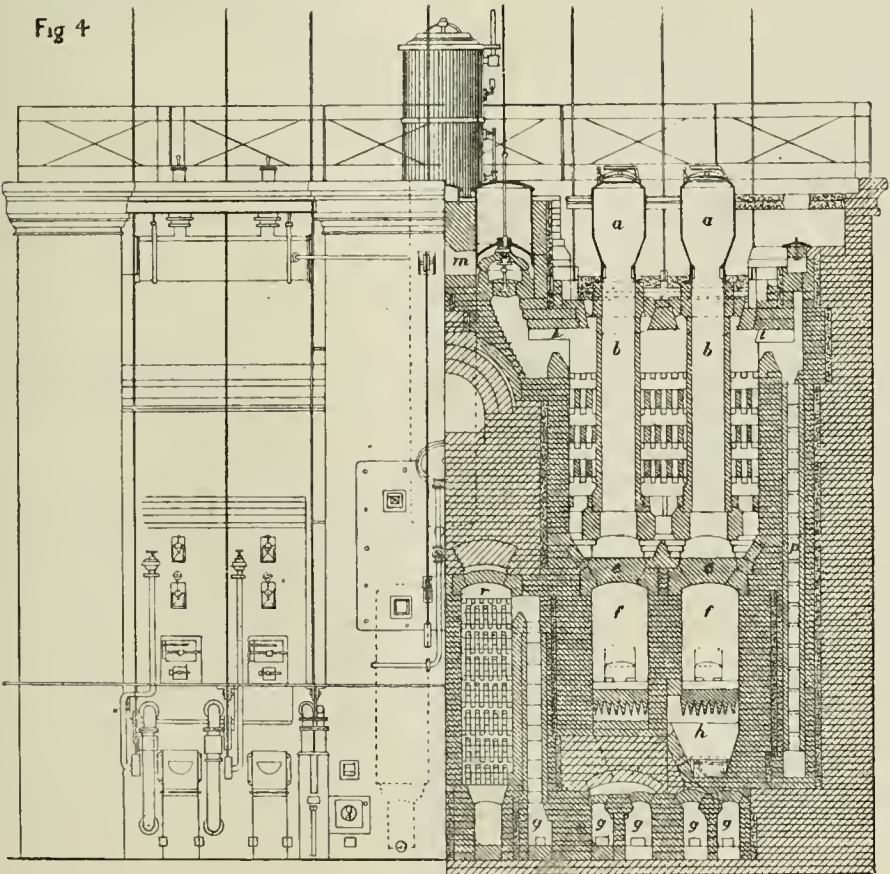
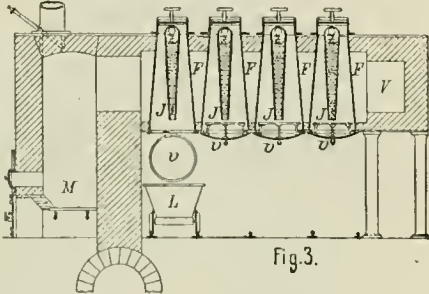
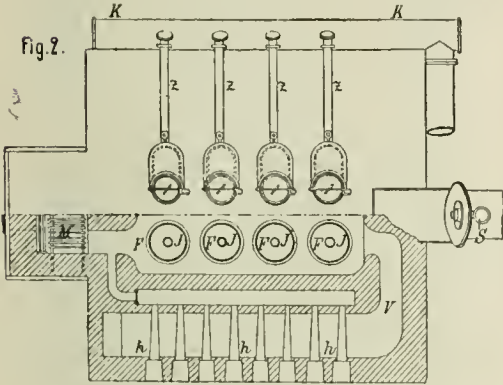
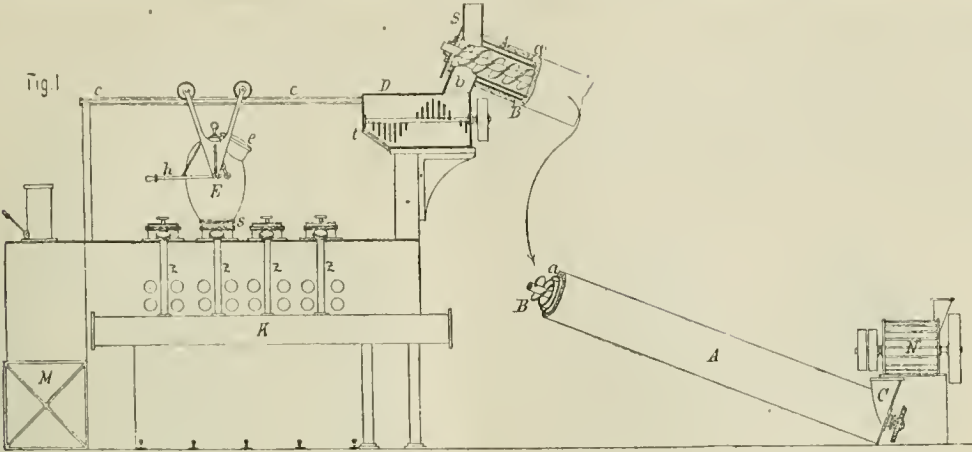
### Journal and Patent Literature.

#### II.—FUEL, GAS, AND LIGHT.

*Improvements in the Manufacture of Illuminating Gas.*  
Dingl. Polyt. Journ. 253 [6] page 233-244.

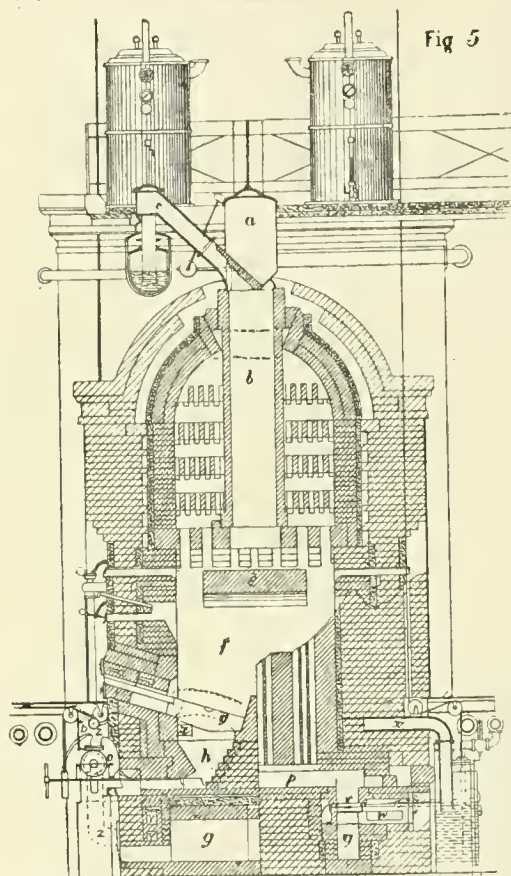
BULL'S Gas, Light, and Coke Co., Liverpool, Germ. Pat. 26093, 1883, propose to use powdered and desulphurized coal for the manufacture of gas. "Coal-duff" is ground in a mill N (figs. 1, 2, 3) and discharged through C into a jacketed cylinder A. An endless screw conveys the coal to the top end of A, where a pipe S allows of the escape of sulphurous gases. Through the pipe b the fuel is emptied into the mixing apparatus D, where it is mixed with tar or naphtha. From here the so prepared coal is emptied through a door t into the vessel E which moves on rails. By the withdrawal of a slide s the coal falls into the retorts F, which are of a conical shape, and set in two parallel rows. Each retort has a neck fitting exactly into the bottom of E; a door v serves for discharging the contents of a retort into bogies L. In the inside of the retorts there is a pipe J tapering towards the bottom, and provided with holes for conducting the gas generated through branches Z to the hydraulic main. Heating gas is produced in the generator M, and the air necessary for combustion enters through pipes h heated by fire-gases. In order to remove as much sulphur as possible from the coal-duff employed in this process, it is first exposed to the air, then washed and powdered. By passing through the heated cylinder A it is dried, and gives off sulphurous acid, which escapes at S. In this manner the fuel is said to reach the mixing apparatus D completely dry and nearly free from sulphur. On account of the latter circumstance the gas needs no purifying, and since no carbon is combined with sulphur the lighting power of the gas is increased.

For the manufacture of lighting and heating gas, B. Andreae, Vienna (Ger. Pat. 26985, 1883), empties coal through a hopper a (figs. 4, 5, 6) into retorts b, where it is subjected to destructive distillation. The gases given off are purified as usually. The coke formed is discharged into the chamber f, lying below, and can be withdrawn as such or converted into water-gas in the same place; or it can be burnt for heating the retorts. In the latter case the combustion gases are made to heat steam boilers, and afterwards pass into a chamber r to give off the greater portion of their heat. To convert the coke in f into water-gas the air openings are shut off and superheated steam is made to meet the incandescent coke.





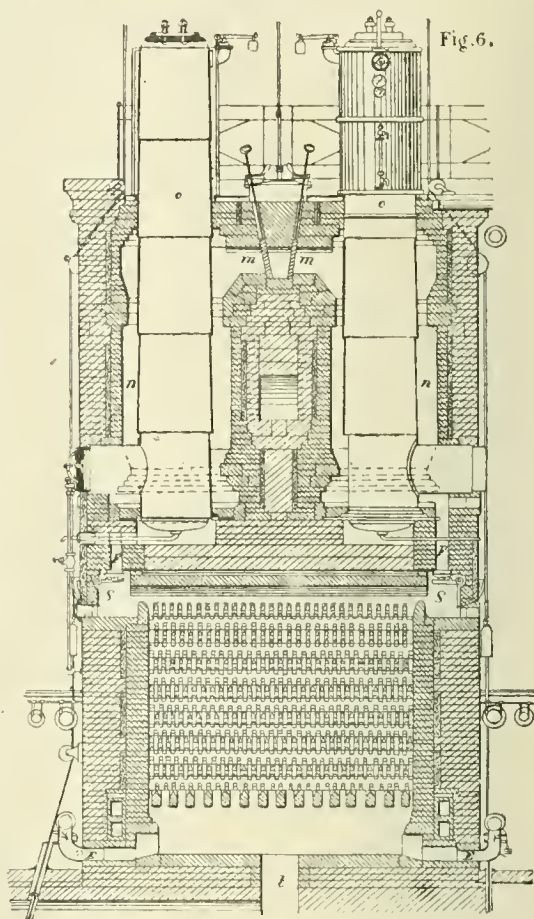
Two such apparatus always work together in one system. While one works with air, burning coke in the usual way, the other converts its coke into water-gas. The steam necessary for this purpose is the exhaust of a steam-engine. In order to carbonize the water-gas it is made to pass through a furnace, in which, at the same time, the carbonizing agent flows on incandescent coke. As there are many valves to be closed and opened at regular intervals the author devised a special engine to do this work.



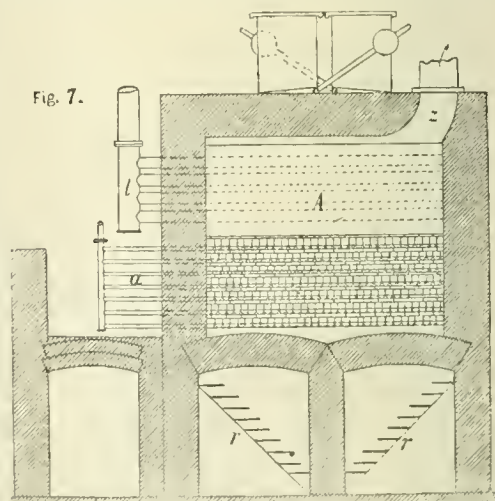
F. C. Glaser, Berlin (Ger. Pat. 26887, 1883), employs for the distillation of peat a furnace consisting of a series of vertical shafts A (figs. 7, 8). The chamber for the combustion of the peat-coke formed is large and very high, in order to give sufficient time to the carbonic acid (produced by the complete combustion on the grates *r*) to be reduced to carbonic oxide by the incandescent peat. The division walls of the shafts are furnished with holes which are in connection with an air-pump to aspirate the carbonic oxide generated in this place, if such be necessary. In the upper part of these walls are larger holes going right through the furnace, which are connected with pipes *l*. In this manner the upper part is kept comparatively cool by a current of air, whereas at the same time hot air is produced without expense. In charging the furnace care must be taken not to bring the fresh peat into contact with hot gases, and should the temperature rise considerably, carbonic oxide must be withdrawn from the lower portion by the air-pump. The draught must also be kept as low as compatible with the proper working. Only in this manner a decomposition of the products of the distillation—ammonia, acetic acid, methyl alcohol, water, tar, etc.—can be prevented. They escape at *Z*, and are conveyed to a scrubber, while the residual gases serve for heating purposes. It is claimed that the heating gas can be produced without any cost whatever, whereas the by-products after paying for working expenses, interest, taxes,

depreciation, etc., still leave a considerable margin for profit, if the process be properly carried out.

H. Hirzel, Leipzig (Ger. Pat. 25909, 1883), uses for the manufacture of oil-gas a spherical retort (fig. 9), which



has only one neck and one opening. The oil enters through the syphon *o*, whereas the gas, instead of escaping through a separate neck, is carried away through C to D.



Th. Foncault, Paris (Ger. Pat. 25730, 1883), has patented an evaporating apparatus (fig. 10) for the manu-

facture of illuminating gas from shale-oils, petroleum, etc. A shaft B holds the fuel, and is protected by a fire-clay ring *a* from being burnt. Holes Z in the lid allow of the escape of the fire-gases into the chimney S. The shaft B is surrounded by another one C, made of cast iron, and provided at its bottom with a casting *c* dipping

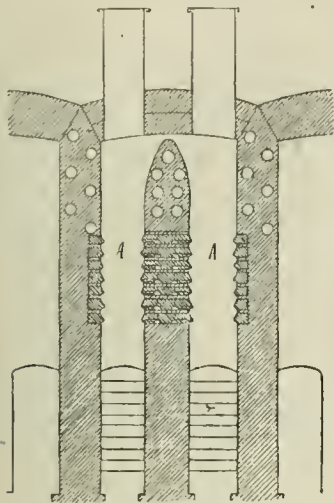


Fig. 8.

in a circular gutter *c*, which holds the oil for evaporation. At the top of C is a deep rim *b*, supporting the bell *d*, which acts as a lute. The space between *f* and C is filled up with charcoal to keep the castings *c* red hot.

of the gas in G, the water and gas receive a circular motion, whereby the gas is thoroughly washed and purified.

A. Binnie, Maori Hill, near Dunedin, New Zealand (Ger. Pat. 23854, 1882), causes a mixture of fat and water to drop on to a red-hot retort. The gas produced is mixed with air, and, after heating, converted into lighting-gas.

M. Gross, New York (Ger. Pat. 25471, 1883), proposes to make illuminating gas from petroleum and superheated steam in retorts open at both ends (figs. 11, 12, 13), whose middle portion is exposed to the full heat of the

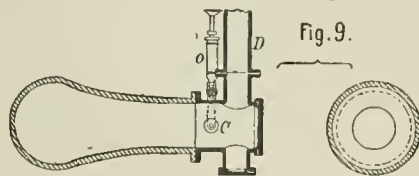


Fig. 9.

furnace. They are partially filled with a porous material, which is always kept at a white heat. A fireclay piece *p* acts a firebridge to direct the gases towards the bottom. One end of the retort is formed by a heavy cast iron plate D provided with a round opening *o* to carry away the gases generated. A cage G formed of iron rods is fastened to G, and holds the porous material, if a combustible stuff, as *f.i.* coke be employed; if the stuff be incombustible the cage G can be dispensed with. Oil enters the retort through an opening in Q, and is made to drip on to the perforated plate *n*. Superheated steam blows from the side just under the bottom of this plate, and mixes intimately with the carburetted vapours. The mixture is aspirated through the white-hot porous

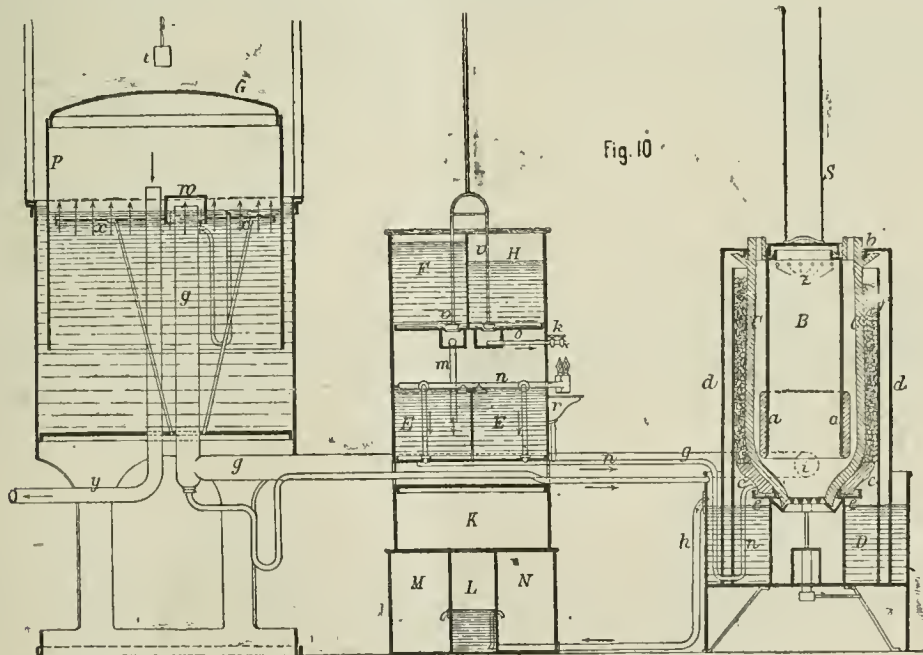


Fig. 10.

The gas produced by the evaporation in *c* travels upwards through the charcoal, then downwards through the flue between *f* and *d*, and through the opening *i* into the pipe *g* to the gasometer. The pipe *n* feeds *c* with oil. Tanks E hold the oil to be evaporated, which is pressed into pipes *n* by water pressure from F. A lever *v* with a counterpoise *t* moving up and down with the gasometer regulates the flow of the oil. The vessel L collects the residues of the condensation, and also separates the oily parts from the water. The gas generated in *c* passes into the regulator G in the direction of the arrows in the figure. Owing to the screw-like openings for the passage

material, and in this manner a combustible gas is formed chiefly consisting of hydrocarbons of the methylene and aethylene group, hydrogen, carbouic oxide, and tar



Fig. 11.

vapours, whereas no nitrogen, nor carbonic acid, nor ammonia, nor sulphur compounds are present.



E. J. Jerzmanowski, New York (Ger. Pat. 27145, 1883), has patented the process of making illuminating gas by carbonizing impure hydrogen, produced by means

then go into the purifier P, and thence to the carbonizers F, which are filled with fire-resisting refractory material, and heated by the combustion of producer-gas. When

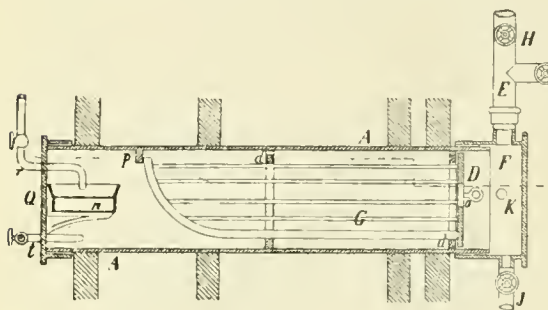


Fig. 12.

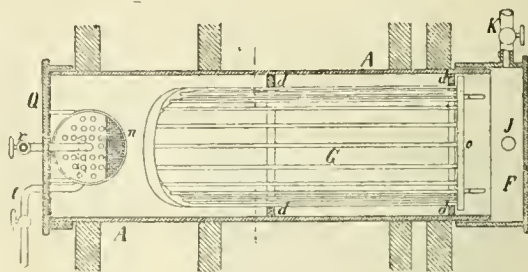


Fig. 13.

of lime. A kiln B (figs. 14, 15) filled with lime is raised to a high temperature by burning producer-gas from the generator A. As soon as a sufficient heat is attained in

F is sufficiently hot the purified gas—chiefly hydrogen—is made to pass through it along with the carbonizing agent. The gas thus carbonized goes to a receiver O,

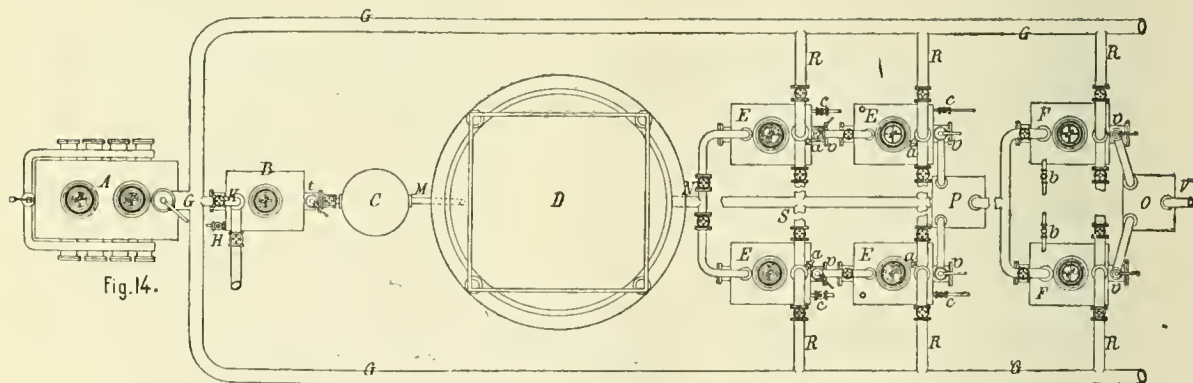


Fig. 14.

B, an injector H blows into B steam and petroleum, which are decomposed chiefly in hydrogen and carbonic acid along with small quantities of carbonic oxide, marsh

and thence to the gasometer. If the absorbers E become inefficient the lime is causticized by heating it in an atmosphere of producer-gas, and air and steam if re-

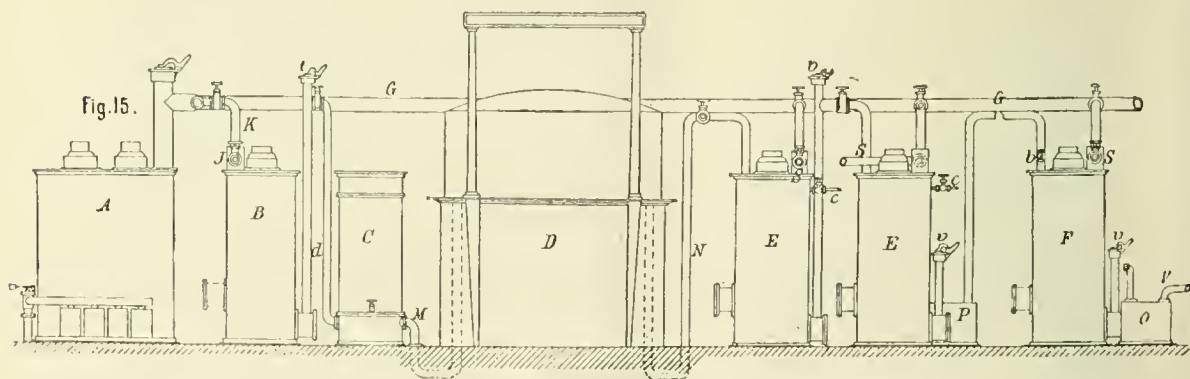


Fig. 15.

gas, and other impurities. The gases pass through a cooler C to the gasometer D, and thence to absorbers E, filled with caustic lime to absorb carbonic acid. They

quired. There are two sets of absorbers, which are used alternately. To dispense with the producer A the lime kiln in fig. 16 is employed. After heating the lime, steam

and a volatile hydrocarbon are injected into the kiln, the gases escaping at *b*. As soon as the heat has sunk under a certain degree the steam is shut off, and air is

the chimney, overheating on their way steam in the pipes *Q*. The products of the distillation are carried away at *a* through pipes *r*. To obtain a greater yield of tar,

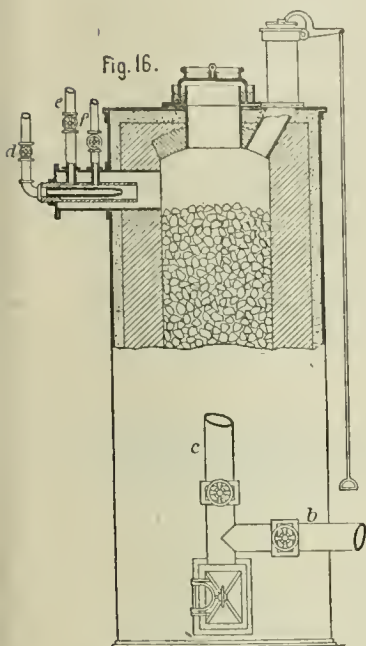


Fig. 16.

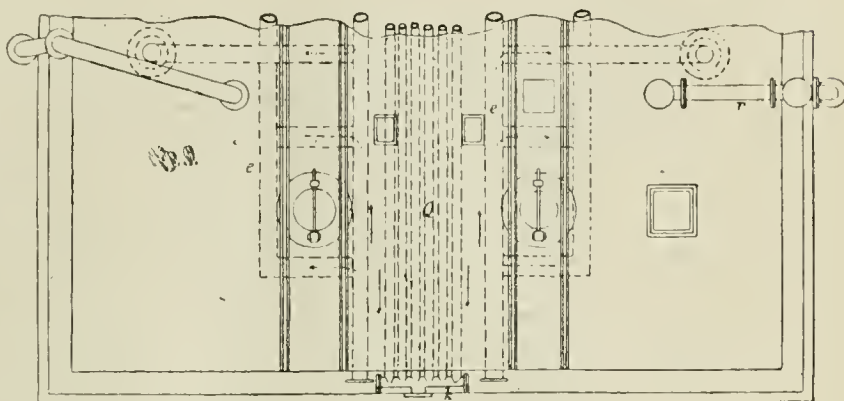


FIG. 2.

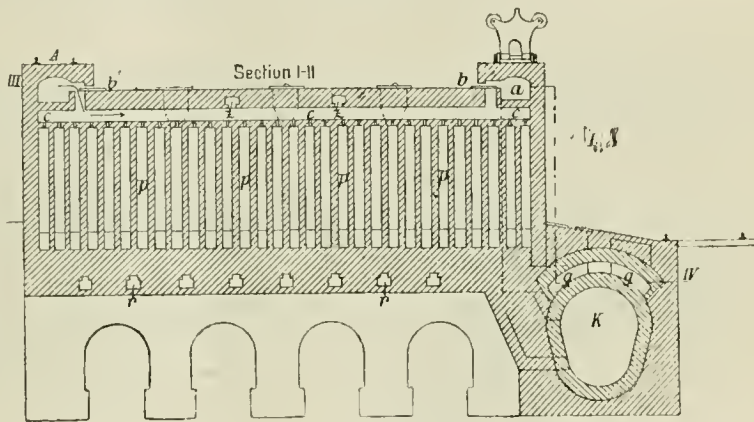


FIG. 3.

allowed to enter through *f*, which causes a combustion of the hydrocarbons, thus restoring heat to the lime.

—S. H.

*Manufacture of Coke and Recovery of By-products.*  
Dingl. Polyt. Journ., 253 [9] 372.

H. STIER, Zwickau (Ger. Pat. 26897, 1883), places two systems of pipes on the top of a coke-oven in order to use the radiating heat for superheating steam, and heating

shale-oil or petroleum is caused to drop on to the incandescent coke. To make use of peat, lignite, and other carbonaceous substances they are heated in the coke chambers to give off at first tar-vapours, ammonia, and an illuminating gas, and afterwards a combustible gas of great heating, but small lighting power. As soon as the temperature is high enough, superheated steam is injected to produce water-gas.

F. Wittenberg, Duisburg (Ger. Pat. 26132, 1883), arranges an air-channel with a slide *b* (figs. 3, 4, 5) inside

Section V-VI

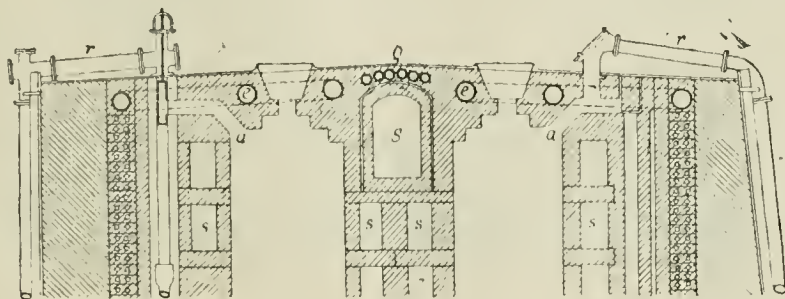


FIG. 1.

the air for combustion. The air enters at *c* (figs. 1, 2) and mixes with producer-gas, and both travel together to flues *s*, heat the coke-oven proper, and then escape to

the top of the furnace. This channel serves, if necessary, to introduce hot air to the main flue *c* and the vertical flues *p*, where the combustion of the coke-oven gases



takes place. There is also a series of other flues which supply the furnace with hot or cold air, as required.

According to O. Ruppert, Gelsenkirchen (Ger. Pat. 26307, 1883), the distribution of the gas in the Coppeé

at the top, where they are burned, whereas the other gases escape through the different flues *a*.

Professor Cl. Winkler discusses in the *Jahrbuch für das Berg und Hüttenwesen im Königreich Sachsen*,

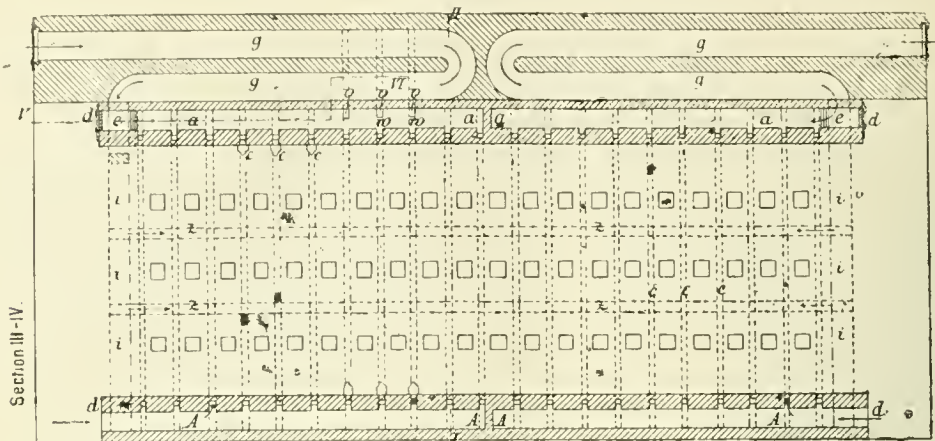


FIG. 4.

oven is very irregular owing to the wide cross-section of the vertical flues, and the unequal distance of the same from the main escape-flue. He therefore proposes to divide the channels in from two to six groups with as

1884, the recovery of ammonia from coke-oven gases. He points out that it is well worth while to make experiments, on not too small a scale, for finding out the conditions under which coke-making can be carried out best.

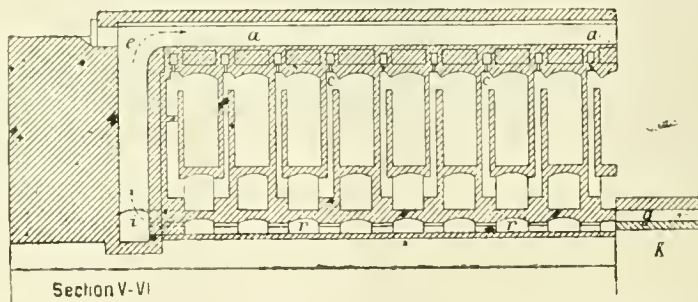


FIG. 5.

many escape-flues *a* (figs. 6, 7, 8), and to regulate the draught of every one of them separately. To heat better that portion of the coal near the doors whereby the yield of the by-products is increased, a hanging bridge *s* (fig. 6)

For this purpose the coking process, and the recovery of the volatile products should be considered as two separate processes. The oven must be heated by an independent producer, and not by gases given off in the coking pro-

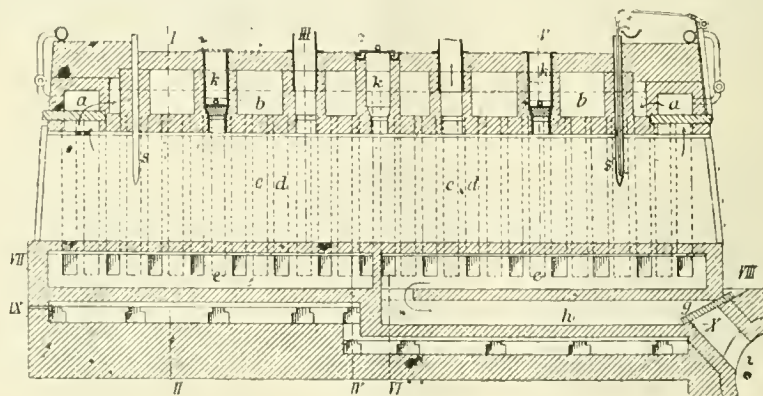


FIG. 6.

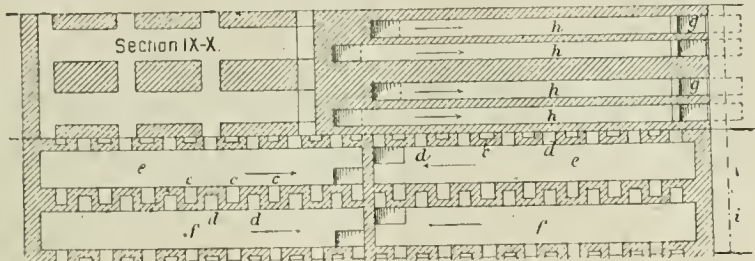
projects into the oven. It is either made of fire-brick, or better still of two iron plates, forming a pocket, which can be cooled by water. The gases from the part of the oven thus separated are directly conveyed to the channels

cess. In this manner we should obtain valuable information: we should get to know what heat is required for the coking process proper, under what conditions the best and densest coke can be made, how to obtain the greatest

yield of it, what is the composition of the volatile products of the distillation, what is the yield of tar and ammonia, what contrivances are required for their condensation, what quantities of water can be boiled down by the gases generated, what is their calorific value after cooling, with what success can they be used for heating other coke-ovens, what influence has the addition of alkalis or chlorine compounds to the coal on the yield of ammonia and so forth. It is well known that not the total nitrogen of the coal can be converted into ammonia.

100 parts of coal therefore gave after coking—

53.2 parts coke			and	46.8 parts volatile products		
Consisting of		Equal to		Consisting of		Equal to
C	39.91 parts	68.3		C	18.53 parts	31.7
H	0.26	6.9		H	3.49	93.1
O	1.27	21.2		O	4.72	78.8
N	0.31	28.7		N	0.77	71.3
S	1.40	72.0		S	0.52	27.1
Ash	10.05	100.0		Ash	—	0.0
Water	—	0.0		Water	18.77	100.0



Section VII-VIII

Fig. 7.

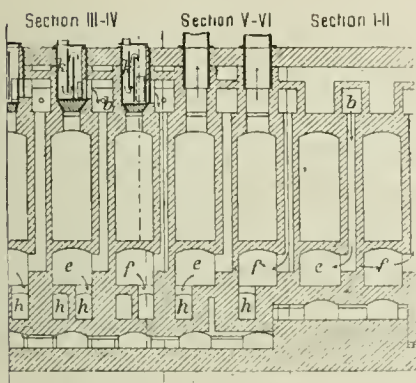


FIG. 8.

W. Foster found in distilling a coal with 1.73 per cent. nitrogen—

11.51 %	of the total nitrogen as ammonia.
1.56 %	" " " cyanogen.
35.26 %	" " " in the gases.
48.66 %	" " " coke.

Winkler examined the details of the process in the coke works at Deuben. The charge, consisting of 4061.5kg. coal had the following composition :—

C	..	..	..	..	58.44 %
H	..	..	..	..	3.75 %
O	..	..	..	..	5.99 %
N	..	..	..	..	1.08 %
S	..	..	..	..	1.92 %
Ash	..	..	..	..	10.05 %
Water	..	..	..	..	18.77 %

100.00

and yielded—

719.5kg.	good coke.
1359.5kg.	cinders.
144.0kg.	coke ash.
2223.0kg.	

An average sample, taken in proportion with these weights, tested :

C	..	..	..	..	72.88 %
H	..	..	..	..	0.48 %
O	..	..	..	..	2.31 %
N	..	..	..	..	0.56 %
S	..	..	..	..	2.56 %
Ash	..	..	..	..	18.36 %
Water	..	..	..	..	2.85 %

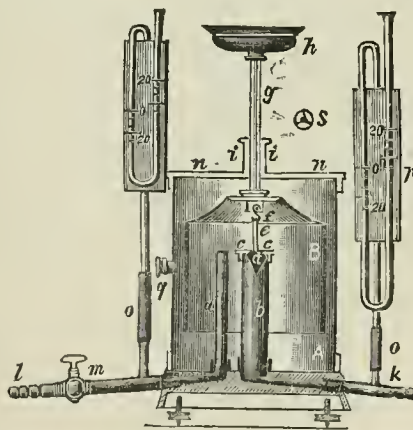
100.00

The author further calculates that 18 million tons of coal are coked annually which would yield 58,600 tons ammonia, that is just as much nitrogen as is used for agricultural purposes in the form of sodium nitrate.

—S. H.

Gas Regulators. R. Muencke. Chem. Zeit. 51, 911.

REGULATORS for ensuring constant gas pressure may be divided into two classes—"dry" (in which a membrane is employed) and "liquid." The latter, on account of their handiness and constancy, are to be preferred for laboratory use. That made by V. Wiesnegg, of Paris, according to Moitessier, is the best; it may readily be taken to pieces, friction on the guides of the bell is minimised, and by means of two manometers the pressures of



the incoming and outgoing gases may be indicated; the author, who has such a regulator in use in his laboratory, considers that it is indispensable where constant pressure is required. The construction of the apparatus is indicated in the figure. The cylinder A, which is levelled by three regulating screws, and is 12 to 14cm. in height, is perforated at the bottom to admit the gas inlet k and outlet l, each of which is provided with a manometer; it is fitted with a cover n, through which passes the bearing rod g. This latter is composed of three strips of brass arranged (as shown with bearing tube i in section at S) to minimise friction by restricting the rubbing surfaces to three lines, carrying the loading pan h above, and the tinned sheet brass bell B below. The bell is connected by e and f to the ball valve d, working in the screw collar c. To use the apparatus, h is unscrewed, the cover n



removed, and the tube *q* is unplugged; a mixture of 2 parts pure glycerine with 1 of water is then poured in (*m* being open) until it overflows at *q*; *n* and *h* and plug *q* are then replaced, and *k* and *l* connected up with the gas main and the burner respectively; the bell being then filled with gas rises, and thus closes the valve *d*, until the gas passing out by *l* causes *B* to fall, and thus the valve is again opened.—W. G. M.

*The Light-giving Power of various Petroleum of Commerce, and the Luminous Efficiency of, and Consumption of Oil in the Commoner Petroleum Lamps.*  
Dr. W. Thörner. Chem. Zeit. 49, 876.

The first series of experiments was conducted with a good microscope lamp with a 15mm. round burner, and a Bunsen's photometer, using Stearine candles; the unit of time was one hour, two observations being taken, the one at the beginning and one at the end of the time, the recorded results being the mean of these.

	Candle Power.	Consumption of Oil per hour.	Candle Power per 20gr. per hour Oilconsumption.
Normal Petroleum I. B.P.=150°–250° C. ....	7–7.5	22g.	6.7
“ “ II. “ =150°–300° .....	7–7.5	21g.	6.9
“ “ I. +20 % Lubricating Oil .....	5.5	19g.	5.8
“ “ I. +40 % “ “ .....	5.0	18g.	5.5
“ “ II. +20 % Petroleum Essence .....	8.5	22g.	7.7
“ “ II. +20 % Lubricating Oil .....	5.0	17g.	5.9

This table indicates that petroleum with B.P. from 150° to 300° C. are best suited for burning purposes, while any addition of lubricating oils (that fraction of raw petroleum boiling above 300°) diminishes the value. Petroleum essence (B.P. below 150° C.) increases luminosity, but also consumption, and adds greatly to the fire risk. In 185 varieties of commercial petroleum examined, only three contained notable quantities of the low boiling oil, while very many contained more or less (up to 34 per cent.) of the lubricating oil. Of 14 samples given in a second table, 5 showed a fair luminosity of 6.5–7 candle power; the others varied in flash test from 22.2° to 33°, and oil consumption from 18gr. to 22.5gr.; one, much adulterated with light oil, gave flash test 14.3, candle power 8.9, oil consumption 21. In the second series a petroleum composed of 8.6 vols. petroleum essence, 64.4 vols. burning oil (150°–300° C., B.P.), and 27 vols. lubricating oil, of flashing point 25.9° C., was used. The results showed that, with all lamps of similar construction, a given increase in oil consumption is accompanied by a corresponding increase in luminosity, and, for a given consumption of oil, a lamp of newer construction gives a better light than an older form. The results are thus tabulated:—

*Regenerative Coke Furnaces.* A. J. Boulton, London.  
Communicated by the “Schlesische Kohlen und Koks-werke,” Gottesberg. Eng. Pat. 5979. Dec. 31, 1883.

UNDER this invention it is proposed to construct furnaces for coking coal, in combination with regenerators, of the type known as “Siemen's regenerators.” The coking furnace is built above regenerators. Prior to the introduction of the coal the interior of the furnace is heated to a red heat by the hot gases from one or other of the generators. Coal is then introduced and distillation commenced; the gaseous products are removed from the coking furnaces either by their own pressure or by mechanical suction and pressure, and after the condensation of the tarry products by dip pipes in a hydraulic main are returned alternately

through one or other of the regenerators, and burnt with air heated by being passed through the hot regenerator, which some time previously acted as a flue to conduct the hot combustion gases to the chimney and has thus become heated. Thus the regenerators act as follows: each becomes alternately a hot gas flue conducting off the hot products of combustion, and are regenerator. Thus a fresh supply of heat is afforded for the continuance of the coking operation. The heat therefore necessary for the process of distillation is almost wholly supplied by the coking operation itself. The currents of gas and air are worked alternately through each pair of regenerators in the usual manner. The arrangement may also be such that the air alone passes through and is heated up by the regenerators. The claims are—(1) The combination of regenerators and coking furnaces. (2) The arrangements for removing the tar prior to the combustion of the gaseous distillate. (3) The combination in (1) and removing the gases by suction or pressure. (4) The combination of regenerators, furnaces, and the heating of the air for the combustion of the gases. (5) Employing alternating currents through the coking furnaces, and the general arrangements of the apparatus.—C. C. H.

LAMP.	Size and variety of Burner.	Candle Power.	Consumption of Oil per hour.	Candle Power per 20gr. consumption per hour.
1. Small Lamp with .....	10mm. flat burner	2–2.5	10.0g.	4.4
2. Lamp without glass chimney .....	7 “ “ “	3.5–4	15.4	5.2
3. Microscope Lamp .....	15 “ circular “	6.5	20.8	6.3
4. Cosmos burner .....	18 “ (10”) “ “	8.0	27.2	6.0
5. “ “ .....	18 “ (10”) “ “	9.0	28.0	6.1
6. “ “ .....	21 “ (11”) “ “	11.8	40.0	6.0
7. “ “ .....	24 “ (11”) “ “	10.5	35.0	6.0
8. Cosmos Reform burner .....	18 “ (10”) “ “	10.0	39.5	6.5
9. “ “ .....	24 “ (14”) “ “	12.5	37.5	6.7
10. Victoria “ .....	18 “ (10”) “ “	10.5	29.5	7.1
11. “ “ .....	24 “ (11”) “ “	17.0	46.0	7.4
12. Sun “ .....	24 “ (11”) “ “	21.0	61.0	7.0
13. Mitrailleur “ .....	12 pieces	19.5	60.0	6.5

*An Improved Regenerative Furnace for the Manufacture of Gas.* J. E. Walsh, Halifax. Communicated by A. Ryex, Ghent. Eng. Pat. 8879. June 12, 1884.

The object sought in this patent is to utilize heat otherwise wasted, and to produce more gas from a given amount of fuel at a less cost. The improvements consist in so lowering the level of the furnace that an eighth retort can be added to the seven usually in use in such furnaces, in making the fire-box of the furnace of a V shaped section, so as to gradually widen at the upper parts in constructing "recuperators" in the side walls of the furnace in such a manner that the air supplied for the consumption of the gases from the furnace passes backwards and forwards, and thus heated emerges at the top of the fire-box, and causes a considerably hotter flame which circulates amongst the retorts. The patentee claims as novel—(1) the shape of the fire-box, and the serpentine passages round the retorts. (2) The construction of the recuperators. (3) The arrangement of gas furnaces, retorts, and recuperators as described.

—C. C. H.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Examination of Russian Petroleum and Petroleum Lamps.* Dr. J. Biel, Dingl. Polyt. Jour., 252, 119.

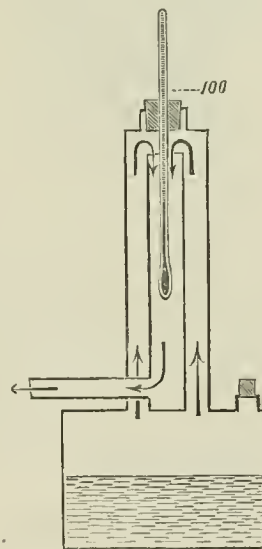
CONSIDERABLE quantities of Russian petroleum are now imported into Germany, and the object of the author's investigation was to determine the characters of different samples of the imported product, and to ascertain what composition petroleum should have, in order to meet the requirements of the consumer as regards cheapness, lighting-power, and freedom from danger. The method of examination used was that recommended by Prof. Engler and Beilstein. The fractional distillation is carried out in a glass flask of 500 c.c. capacity, in which are put 250 grams. of petroleum. The flask is tightly wrapped round with thin brass gauze or glasswool, to protect it from too rapid changes of temperature, and it is connected by a Glinzky's dephlegmator with a Liebig's condenser. The thermometer is so fixed in the dephlegmator that the mercury-bulb is on a level with the exit-opening. The distillation of the light hydrocarbons first takes place, up to a temperature of 150°, and it is ended when not more than 10 drops come over in the course of a minute. With a little practice it is easy to see when the distillation must be interrupted, as the thermometer, having reached 150°, begins to fall rapidly. At the commencement, the flame should not be larger than is necessary to drive over the light oils. The distillate so obtained is received in a tarred vessel and weighed. The normal petroleum, that is to say, the hydrocarbon with a boiling-point of 150-270°, next comes over, the flame being gradually enlarged. It is easily seen when the distillation is finished, because the thermometer then no longer remains stationary at 270°. This distillate also is received in a tarred flask and weighed. The weight of the heavy oils is ascertained by subtracting the weight of the cleansed flask and dephlegmator from that which they possess immediately after the distillation of the normal petroleum. Five samples of petroleum, examined by this method, furnished the following results:—

	Petroleum sample.					Pyronaphtha.
	A.	B.	C.	D.	E.	
Specific gravity.....	0·820	0·820	0·835	0·857	0·867	
Temperature at which inflammable vapours were given off.....	52·5°	35°	41·5°	67·5°	91°	
Distillate at 150°.....	0	8	10	6	0	0
" from 150°-250° ..	92	76·5	63·5	44·5	30·5	
Remainder .....	7·2	13·5	30·5	55·5	69·5	

It will be seen that the temperatures at which these samples gave off inflammable vapours were considerably higher than the temperature taken as sufficient in Germany. The author concludes his paper with a tabular statement of the results of some photometric comparisons of these petroleum, burnt in three different kinds of lamp-burners.—E. G. C.

*On the Mode of Conducting Fractional Distillation for the Purpose of Determining the value of Chemical Products.* G. Lunge. Chem. Ind., 7, 150.

IT is a well-known fact that for a large group of organic substances, including a number of commercial products of great importance, fractional distillation is the only method available for determining their value. Unfortunately this method is not adapted for the separation of the various constituents of a mixture, and in most cases affords only an empirical expedient by which the quality of these substances is judged. Thus 90 per cent. benzol does not contain 90 per cent. of pure benzene, but is a product of which 90 per cent. by volume distils under 100°. To ascertain what fractional methods were actually employed the author, who is interested in the question of bringing about an international system of analysis, submitted a series of questions to the committee of the German society, and obtained the following information. Only a few members of the fatty group are valued by fractional distillation, e.g., alcohol, methyl alcohol, glacial acetic acid, aldehyde, crude petroleum, benzene, and burning oil. However, the fractional distillation of aromatic compounds is largely practised, e.g., benzenes (toluene, xylene) nitrobenzenes, etc., aniline oil (aniline, toluidine, xylydine, cumidine) dimethyl aniline, diethyl aniline, benzyl chloride, benzotrichloride, benzaldehyde, quinoline, quinaldine, earbolic acid, naphthalene, etc. With regard to the form of apparatus it was ascertained that the German tar distillers do not employ the retorts used in England with the thermometer immersed in the liquid. Retorts are used by some manufacturers for the high boiling portions of petroleum, naphthalene, etc., but in most cases fractionating flasks with or without the dephlegmation of the vapours.\* In discussing the position and shape of the thermometer, the author refers to an apparatus employed by a large firm for ascertaining the 100° point. It consists of a tin vessel fitted with a cover on which a double cylinder is placed (see figure). The



vapours of the boiling water stream into the intermediate annular space, and pass into the inner cylinder which they enter at the top and leave at the bottom as shown by the position of the arrows in the accompanying drawing. The thermometer is placed in the inner cylinder, so that the 100° mark is situated immediately above the outside of the cork. It is necessary to observe the

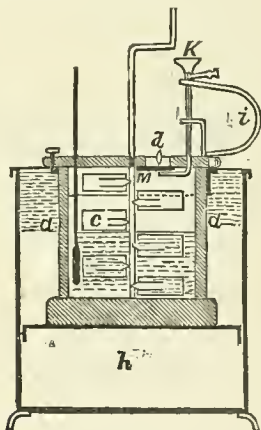
\*Note by Abstractor.—It may be mentioned that although English naphtha is still valued by distilling from an ordinary retort, and determining the volume percentage of distillate obtained at a temperature not exceeding 120°, most English tar distillers ascertain the percentage composition by distilling the naphtha in a flask fitted with a three bulbed apparatus used for fractional distillation.



barometric pressure before making this test, and to make the necessary correction if required. As to the position of the thermometer it is generally excepted that its gradations should be as far as possible within the vapour. 90 per cent. and 50 per cent. benzenes should not begin to distil under 80°. In the case of 90 per cent. benzenes nothing should distil above 120°, and with 50 per cent. benzene nothing above 130°. Naphthalene is distilled in a retort, and the distillate boiling between 210° and 225° collected. Benzaldehyde is distilled in a current of carbonic anhydride. In the case of methyl alcohol 98.5 to 99 per cent. should distil between 66° and 66.5°. Petroleum which gives more than five per cent. distilling under 150°, or not more than 90 per cent. at 300° is objectionable. Good petroleum should distil between 150° and 300°.—D. B.

*Flash Point Apparatus for Petroleum.* By E. Leybold's successor. *Chemisches Centralblatt*, 15 425 (German Patent).

THE disadvantage of the petroleum vapours diffusing gradually from below upwards is got over by having a powerful stirrer *c* kept constantly moving till the moment of explosion, and which thoroughly mixes up the whole of the air and petroleum vapours which are above the oil level in the vessel *a*, and the air may bear a fixed volume relation to the oil, *e.g.* 1 : 1.



By this arrangement there is the further advantage that the position of the igniting flame is of no consequence, and the size of the heating flame and the size and initial temperature of the water-bath *h* are without influence on the result. The igniting flame *d* is not brought with its point into the vapour mixture, but by pressing with the hand on the knob *h* is lowered into the gaseous mixture. The heat from the flame radiating downwards is stopped by the plate *m*, which sinks with the flame, the hot products of combustion escaping through the opening thus made in the cover.—F. L. T.

*Obtaining Oil and Gas from Minerals, &c.* N. McFarlane Henderson, Broxburn, N.B. Eng. Pat. 5873, December 27, 1883.

THIS specification describes certain improvements in the process and apparatus specified in Patent No. 1327 of April 10th, 1873, for the purpose of obtaining oil from minerals. Rectangular vertical retorts are constructed in two rows forming a bench; between them are flue spaces communicating with each other. The retorts are provided with sloping bottoms, the usual steam inlet pipes and outlet pipes, for the products of distillation communicating with the suitable condensers. Two regenerative chambers, similar to those employed in a Siemens furnace, are formed underneath the retorts, and are in connection with the flues. Gaseous fuel is used, burned only in one of two sets of combustion chambers, and after heating, the retorts pass through the regenerator on its way to the chimney in the manner usual in a Siemens furnace,

the currents being alternately reversed through each side of the apparatus. Each pair of retorts are in communication at their upper parts, so that the retort undergoing the second stage of distillation being cut off from the main leading to the condensers gives off its gases through the charge of the other retort in its first stage, the products of distillation passing downwards, and thus away to the condensers by the outlet pipe at the lower part of the retort. Modifications of the older form of apparatus, so as to work in a similar manner to the above, are also described. The patentee claims:—(1) Treating minerals at different stages in contiguous retorts as described. (2) The combination of apparatus. (3) The combination of the apparatus modifying the older form of apparatus. (4) Obtaining oil from minerals by means of the apparatus described in claim (2).—C. C. H.

*Obtaining Oil etc., from Minerals, &c.* J. McCulloch and H. Macvicar, Calderbank, Lanark. Eng. Pat. 5903, December 28, 1883.

TO effect the distillation of oil and other products from coal and minerals in a more economical and better manner than that hitherto pursued, the apparatus described below is employed. Vertical retorts are formed in the interior of a tall cylindrical furnace, by means of two concentric walls intersected by radial walls, thus forming a series of segmental cells fifty feet long or upwards. The spaces between the segmental retorts so formed communicate at their lower ends in the combustion chambers burning gas or other fuel, and at their tops with a central inner shaft communicating with a chimney. The material submitted to destructive distillation is fed in at the top through hoppers, and during its gradual passage downwards is submitted to an increasing heat circulating round the retorts from the combustion chambers. As the heat increases the less volatile constituents are expelled, and finally by the admission of steam at the bottom part of each retort, ammonia can be obtained prior to the removal of the spent mineral by means of a screw creeper. The products of distillation can be collected from the tops of the retorts and condensed, or may be separately collected from different openings in each retort. The patentees claim:—(1) The process of subjecting minerals to distillation in long vertical retorts 40 feet high and upwards, as described. (2) The construction of heating flues and retorts as described.—C. C. H.

#### IV.—COLOURING MATTERS AND DYES.

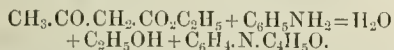
*On Quinoline and Pyridine Compounds.* Dingl. Polytech. Journ., 252, 123.

IN the manufacture of yellow colouring matters from pyridine and quinoline bases, phthalic anhydride can, according to E. Jacobsen, of Berlin, be used instead of chlorinated phthalic acids. The yellow colouring matter obtained by treating that portion of the coal-tar bases boiling at from 235° to 240° with phthalic acid is identical in composition and characters with the colouring matter formed from quinaldine and phthalic acid. The same colouring matter is also found to result from the reduction of ortho-nitrobenzylideneacetone (methylquinoline or quinaldine). The coal-tar bases boiling between 240° and 310°, which likewise yield yellow colouring matters with phthalic anhydride, appear to contain compounds homologous with quinaldine. Yellow colours are also obtained when cumidine is converted by the action of aldehyde and hydrochloric acid into cumoquinoline or trimethylquinaldine (Döbner and Miller), and the latter heated with phthalic anhydride, phthalimide, nitrophthalic acid, or chlorinated phthalic acid. Pure quinoline, synthetically prepared, as well as that from tar, yields no yellow colour with phthalic acid; but on the other hand, yellow colours are obtained if the quinoline be methylated, ethylated or amylated, according to the usual methods, and the resulting products be treated with phthalic anhydride or substituted phthalic acids. The homologues of quinoline, obtained by the action of glycerol and sulphuric acid on toluidine and xyldine, behave analogously. In the production of oxyquinolines,

sulphonic acids of primary aromatic amines are heated together with glycerol and sulphuric acid, according to Skraup's method for the synthesis of quinoline.

For the preparation of oxyquinoline, for example, 100 parts of sulphanilic acid, 120 parts of glycerol, 150 parts of sulphuric acid and 40 to 50 parts of nitrobenzene are heated together. On completion of the reaction the mass is mixed with lime or baryta to remove the sulphuric acid, the next step being a fusion with 2 to 3 parts of caustic soda, the sodium salt of the quinoline-sulphonic acid formed being produced. The mass is now dissolved in water, neutralised with acid, and the separated oxyquinoline purified by distillation or crystallisation. The quinoline-sulphonic acid first obtained is extremely soluble in water and forms colourless needles: the oxyquinoline obtained from it fuses at 192° and is identical with the paraoxyquinoline obtained by Skraup from para-amidophenol.

To prepare oxynaphthoquinoline, 100 parts of  $\alpha$ -naphthylamine-sulphonic acid, 200 parts of glycerol, 200 parts of sulphuric acid, and 50 parts of nitrobenzene are heated for from 4 to 6 hours in an oil-bath at 140° to 160°, at the end of the reaction, the  $\alpha$ -naphthoquinoline-sulphonic acid is dissolved out with water, freed from unaltered  $\alpha$ -naphthylamine-sulphonic acid by heating with a dilute solution of acid chromate of potassium, and purified by repeated solution in soda and precipitation with acid. It is difficultly soluble in water and crystallises from hot water in colourless needles. Fused with sodium hydrate, it yields an oxynaphthoquinoline melting at 270° to 275°. In like manner, instead of sulphanilic acid and naphthylamine-sulphonic acid, orthotoluidine-metasulphonic acid, paratoluidine-ortho-sulphonic acid can in the first place be employed, the resulting products being orthotoluidine-quinoline-parasulphonic acid and paratoluidine-quinoline-metasulphonic acid; from these, by fusion with alkali, paraoxyorthotoluidine-quinoline and metaoxyparatoluidine-quinoline are obtained. As means of oxidation, not only nitrobenzene, but nitrotoluene, nitroxyline, etc., may be employed. The sulphuric acid can evidently be replaced by other substances having an affinity for water. According to L. Knorr, quinoline derivatives, in which the pyridine nucleus is hydroxylised, result from acetoacetic ether and its various substitution derivatives, on the one hand, and aromatic monamines, such as aniline, toluidine, xylidine, cumidine, naphthylamine, amidoanthracene, amino benzoic acid, or their substitution-products (chloro-, bromo-, iodo-, methyl-, ethyl-), as well as their azo-derivatives, on the other hand, when equivalent quantities are heated for many hours to 120° (best in absence of air) with a condensation-agent, as, for instance, concentrated sulphuric acid, according to the equation:—



The substituted oxyquinolines so obtained are stable, difficultly soluble in water, and possess acid, as well as feebly basic properties: they will serve for the manufacture of dyes, etc. Aniline and acetoacetic ether, for example, in the proportions of their molecular weights, are heated to about 120°; preferably in the absence of air. The product is either digested for a long time with concentrated sulphuric acid, or heated with the same acid for a short time to 180°. After dilution with water, the mixture is exactly neutralised with alkali; the oxymethylquinoline, precipitated thereby in flocculi, fuses at 221°.

A. Ladenburg and C. F. Roth (Ber. Deutsch. Chem. Ges., 1884, 513) have shown that piperidine synthetically prepared from pyridine is identical with the base obtained from pepper. W. Königs and R. Geigy (ibid., 589) describe the production of oxypyridine-monocarboxylic acid from oxyquinoline acid, and the formation of pyridine-disulphonic acid by heating piperidine with concentrated sulphuric acid. By treatment of the same with pentachloride of phosphorus trichlor-pyridine is obtained, which gives a crystalline precipitate with mercuric chloride. Pyridine itself in very dilute aqueous solution (e.g., 0.001), yields with chloride of mercury a somewhat insoluble compound,

which dissolves extremely easily in warm water and separates out, on the solution being cooled, in long white needles; a similar solution gives, with cadmium chloride, after some hours' standing, a compound crystallising in long colourless needles, and behaving like the mercury compound as regards solubility in water. Pyridine also yields with mercuric iodide a compound crystallising from hot alcohol in beautiful white needles. Phosphotungstic acid throws down a very difficultly soluble precipitate in acid solutions of pyridine.

According to L. Knorr to obtain phenyl- $\beta$ -imido-butyric acid, a mixture of equivalent molecules of aniline and acetoacetic ether is heated from four to five hours in a sealed tube, at from 150° to 160°. The product is freed on the waterbath from the alcohol formed, the crystals are separated from the mother-liquor by washing with benzene liquor and they can be obtained in a pure condition by a single crystallisation from benzene. By the long-continued action of strong sulphuric acid, in the cold, they are converted into  $\gamma$ -oxyquinoline. The same condensation is effected by prolonged heating in a tube to 120° with 30 per cent. hydrochloric acid. A clear solution of the substance in strong alkali thickens and becomes oily on heating. The oil formed is easily soluble in ether, and smells like aniline, but does not give the chloride of lime reaction. The homologues of aniline behave similarly with acetoacetic ether. Equivalent molecules of orthotoluidine and acetoacetic ether are heated in a tube to 150°. The orthotolyl- $\beta$ -imido-butyric acid crystallises after the removal of the alcohol in beautiful flat needles. By the action of concentrated sulphuric acid this is changed into ortho-tolu- $\gamma$ -oxyquinoline, which exhibits the same characters as  $\gamma$ -oxyquinoline. Acetoacetic ether and  $\beta$ -naphthylamine give at 150° to 180° the naphthalid of  $\beta$ -naphthyl- $\beta$ -imido-butyric acid, and this on treatment with hydrochloric acid yields  $\beta$ -naphthyl- $\beta$ -imido-butyric acid. This is also produced by condensation of  $\beta$ -naphthylamine and acetoacetic ether with its naphthalid, and is obtained from the mother-liquor by evaporation and crystallisation. The acid is dissolved in concentrated hydrochloric acid, and on heating to boiling there is a sudden separation of fine needles, consisting of a hydrochlorate, which is decomposed by washing with water. The residual base is obtained by crystallisation from alcohol in flat, glistening needles, melting at 286°, and possessing the composition of  $\beta$ -naphtho- $\gamma$ -oxyquinoline. This can also be prepared directly from the naphthalid of melting point 200° by heating with strong hydrochloric acid. On heating it with twenty times its weight of zinc dust, one obtains an oily distillate which solidifies into crystalline needles after some time. These are dissolved in dilute sulphuric acid, and on the addition of potassium chromate, a greasy, brown chromate is precipitated, which crystallises from water. The base is freed from the chromate by ammonia, crystallised from ether, and so obtained as a brilliant crystalline mass of melting point 88°, and possessing all the characters of a naphtho-chinoline. The concentrated acid solution fluoresces green, and when diluted dark blue.—E. G. C.

*The Manufacture of New Colour-yielding Products, and of Colouring Matters therefrom.* Dr. Leo. Gans, of Frankfurt, and Dr. Meinhard Hoffmann, of Mankur, near Frankfurt, Germany. Eng. Pat. 816. January 5, 1884.

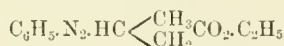
THE inventors claim the discovery of a new disulphonic acid of  $\beta$ -naphthol, which they designate the gamma-disulphonic acid. In order to prepare this acid, the well-known  $\beta$ -naphthol- $\alpha$ -monosulphonic acid is taken as the starting point. The potassium salt of this last-named acid is mixed with three times its weight of sulphuric acid (66° B.), and heated to 40°–50° C. for five to six hours. The same result can also be arrived at by heating the mixture at 100° C. for one or two hours, or by leaving it at 25° C. for eight or ten days. The gamma acid can also be prepared directly from  $\beta$ -naphthol by mixing the latter with five parts sulphuric acid



(66° B.), and cooling down to 0° C. so as to form naphthylsulphuric acid; the temperature of the mixture is then gradually raised to 60° C. during thirty-six hours. The gamma disulphonic acid is by this means almost entirely formed. Schaeffer's monosulphonic acid being produced in small quantity as a bye-product. Other processes are described, all depending upon variations in the time, temperature, and amount of sulphuric acid, but whichever process is adopted, the operation is not considered complete till the melt shows no further trace of the  $\beta$ -naphthol- $\alpha$ -acid. The separation of the acids not required is effected by fractional crystallisation of the salts of Ba, Na, or K, or by partial precipitation with diazo-compounds, such as  $\alpha$ -C<sub>10</sub>H<sub>7</sub>.N<sub>2</sub>.Cl. The new gamma-disulphonic acid can also be obtained from  $\beta$ -naphthylamine by converting this base into a disulphonic acid, diazotising, and boiling with water in the usual way. The gamma-acid is utilised for the production of azo-colours by combining it with the diazo-salts of the amines, amidoazo-compounds, etc. Four of these compounds are mentioned as examples, viz., (1) a yellow colour from diazometanitriline, and the new acid (in alkaline solution); (2) an orange from diazobenzene chloride; (3) a scarlet (Ponceau 3 R) from diazo- $\alpha$ -naphthylamine sulphonic acid; and (4) a cotton scarlet from diazotised amidoazobenzene. The inventors claim also the  $\beta$ -naphthylamine disulphonic acid described, and the series of azo-colours obtainable therefrom, especially mentioning the violet compound produced by diazotising this acid, and combining it with  $\alpha$ -naphthylamine in acid solution, and also the scarlet resulting from the action of its diazo-compound upon  $\beta$ -naphthol- $\alpha$ -disulphonic acid in alkaline solution. — R. M.

*A Manufacture of Oxypyrazols or Substances formed by condensing Hydrazines with the Ethers of Carboketonic Acids.* Charles Denton Abel. Communicated by the "Farbwerke, vormals Meister, Lucius and Brüning," of Höchst-am-Main, Germany. Eng. Pat. 3097. February 11, 1884.

E. FISCHER'S general reaction (Ber. d. dent. Chem. Gesell., vol. xvi., p. 5) forms the basis of the present invention. Acetoacetic ether combines with phenylhydrazine with the elimination of water forming a condensation product of the formula:—



This product is a yellow oil, which, when heated, undergoes further condensation, alcohol being eliminated, and methylphenyl-oxypyrazol being formed:—



This conversion takes place at 100° C., and the methylphenyloxy-pyrazol is obtained as a crystalline solid. It forms alkyl derivatives with the chlorides, bromides, etc., of the alcohol radicals, and with excess of phenylhydrazine, a body of the formula C<sub>20</sub>H<sub>18</sub>N<sub>4</sub>O<sub>2</sub>, dimethylphenyloxy-pyrazol. The corresponding compounds from the hydrazines of other bases are mentioned. — R. M.

#### ERRATUM.

In J. S. C. I., October 29, 1884, page 515, column 2, five lines from bottom, read *Morin* for *Isturin*.

### VL.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Process for Bleaching Fibrous Materials.* H. Köchlin. Chem. Centr. Bl. 1884, 688.

It is proposed to bleach fibrous materials by soaking them in alkalis, or caustic alkaline earths, in the presence of sodium sulphite, or hydrogen sodium sulphite, and steaming them, or subjecting them to a current of hot air. — D. B.

*Improvements in Dyeing Mixed Cotton and Silk Lace and other Fabrics.* W. Clarke and H. J. Tansley, Nottingham. Eng. Pat. 7869. May 17, 1884.

In order to dye a mixed fabric, composed of cotton and silk, equally as well as if each had been separately dyed, and to secure difference in tint of the same colour in the different portions of the fabric, this invention proceeds as follows:—The fabric is boiled in a soap solution, such as is used for dyeing silk alone, after which, and the removal of the goods, the solution is neutralized with an acid so as to be only feebly alkaline; if the bath is rendered acid the cotton remains unaffected by the dye in the subsequent operation. The fabric is then immersed in the same bath two or three times, raised to the boiling point, and the dyeing material added by degrees at each successive operation. The silk portion takes a darker tint from the dye than the cotton, and thus the pattern appears upon a dark or light ground, according as the silk or cotton form the ground on the pattern. The patentees claim the method of dyeing mixed goods as described. — C. C. H.

*A Method of, and an Apparatus for, Washing, Dyeing, Bleaching, Rinsing, Impregnating, and Dyeing Fibrous Materials.* W. Scheidt, Kettwig, Prussia. Eng. Pat. 6805. April 25, 1884.

This invention is for a method and apparatus for carrying into effect the various operations connected with dyeing and washing, etc., set forth in the title. The apparatus consists of a cylinder closed at both ends, its interior being provided with perforated diaphragms, the lower one fixed, the upper one movable, between which the material to be operated upon is placed. A force pump is connected to the upper and lower part of the apparatus by pipes and three-way cocks, so that a current of liquid of any kind can be forced through the apparatus in either direction, and finally drawn from the interior of the vessel into a store tank. The specification states that by thus forcing, under pressure, any dyeing or washing liquor through the material operated upon in the cylinder, and by alternating the direction of the flow, the operations can be more quickly carried into effect, and with a more uniform result. Two other modifications of the apparatus are described as adapted for dyeing or washing a web of cloth, or yarns. The whole invention and apparatus is covered by six distinct claims. — C. C. H.

### VII.—ACIDS, ALKALIS, SALTS.

*A Description of H. Grouven's Furnace for the Preparation of pure Carbonic Acid from Limestone, Dolomite or Strontianite, with the Aid of Red-hot Steam.* E. Meyer-Mülsen. Dingl. Polyt. Jour. 252, 68, 1884.

It has already been observed by several chemists that if hot steam be caused to traverse lumps of limestone, dolomite, witherite or strontianite, contained in a tube heated to moderate redness, carbonic acid is given off, and the above-mentioned carbonates are converted into the corresponding oxides. Taking advantage of this reaction, H. Grouven (Ger. Pat. 26248, December 4, 1883), has devised the furnace here described, for the purpose of obtaining carbonic acid contaminated with practically nothing else than aqueous vapour. In figs. 1 and 2 are shown the vertical and horizontal sections respectively of this furnace. To contain the carbonate which is to undergo decomposition seven retorts placed vertically and arranged in a concentric manner are provided. The diameter of these retorts is most advantageously 0.25m., and their length such that from 3 to 4m. come within the heating chamber. Such a battery of retorts suffices for the decomposition of 7.5 tons of limestone per diem, but the number of retorts may easily be increased to twelve or even sixteen, with a proportionate increase in the capabilities of the furnace. The diameter of the retorts, however, should not be increased beyond the size named,

as otherwise the difficulties of thoroughly heating their contents become enormously increased, and from the higher temperature requisite at the exterior the life of the retort is considerably shortened. The retorts are heated by the combustion of a mixture of generator gas and air. The former proceeds directly from the generator, and is forced by means of a Root's blower through the ten openings of the two gas rings to enter the heating or com-

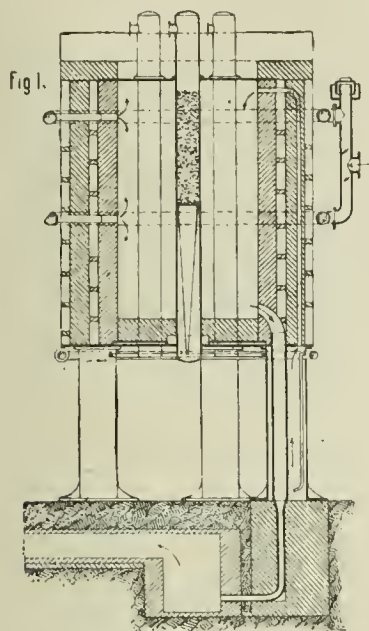
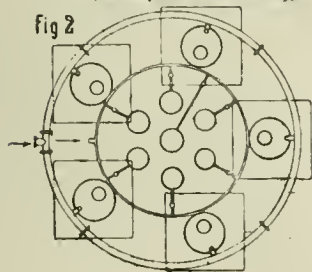


Fig. 2

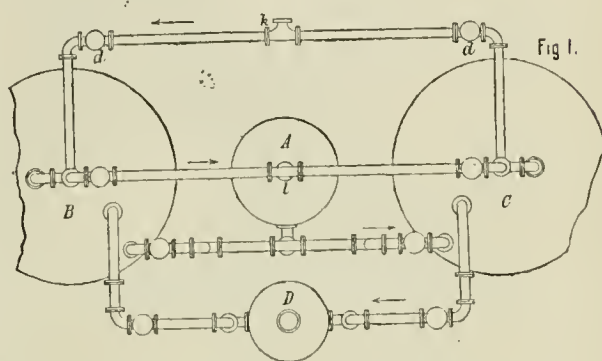


bustion chamber at a temperature of from 600° to 800° C. Atmospheric air heated to from 300° to 400° C. by passing along the five pillars which support the furnace is forced by the Root's blower to enter the combustion chamber through the five tuyeres at the top of the pillars. These currents of air do not meet the streams of gas; no tongues of flame are formed; the mixture of air and gas takes place gradually and uniformly throughout the extent of the combustion chamber, and possible danger to the retorts, owing to irregular heating, is hereby obviated. The upper half of the retorts are filled with the carbonate which is to be decomposed, broken into pieces of from 20 to 40 mm., and the mouth of the retort closed gas-tight by a Morton's door. The retorts are then heated to moderate redness, and steam is introduced at their lower extremity. By the time the steam reaches the movable grating upon which the carbonate rests, it is raised to such a temperature that it readily reacts with the heated carbonate. A steady current of steam is maintained, and the evolution of carbonic acid proceeds with regularity. The gas and aqueous vapour pass out through the side openings at the top of the retorts, and on the condensation of the water vapour the carbonic acid is obtained in a very pure condition. About four hours are required for the thorough decomposition of the contents of these retorts.

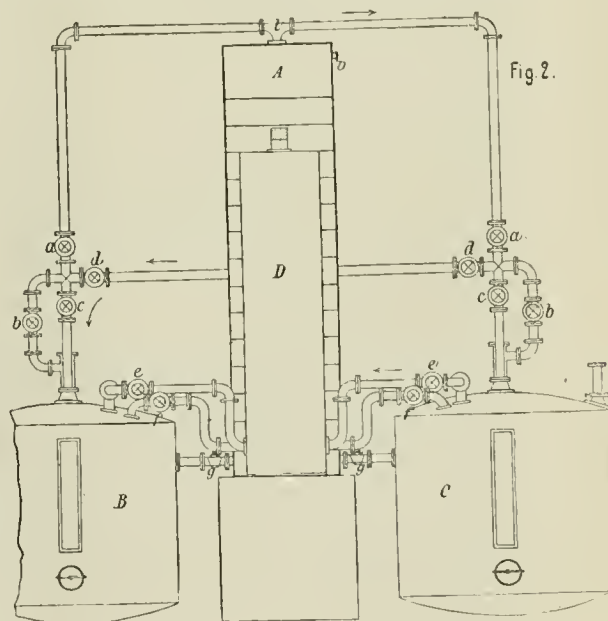
The causticized residue is removed by elevating the movable rests, and the retorts may then be replenished, and a second operation commenced. That this furnace is economical in its working appears from the fact that on one occasion 100 kilos of Rüdeshimer shell-lime were decomposed with a consumption of 12 kilos of coke, and 24 kilos of steam. This furnace may also be applied to the desulphurization of pyrites. In this case sulphuretted hydrogen is evolved, and an impure ferrous-ferrie oxide obtained, as a residue in the retorts.—W. D. B.

# Apparatus for the Production of Ammonia-Soda. Dingl. Polyt. Journ., 252,118.

IN order to ensure an uninterrupted working of the ammonia-soda apparatus, E. Strassburger and P. Fraenkron combine with it the reservoir A (see figure.) When



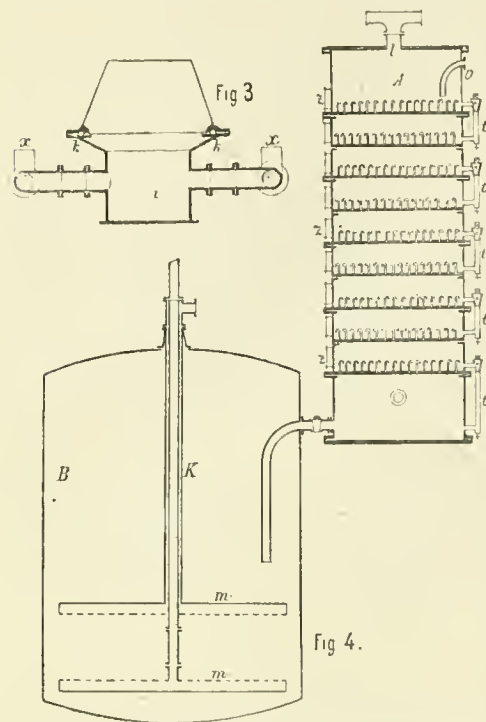
the reservoir C is filled with fluid coming from the apparatus A, the product in the reservoir B is ready. No pipes are employed to cool the bicarbonate, but it is worked with carbonic acid cooled down to the required temperature. This is more satisfactory, as the pipes sometimes get



damaged, and water in consequence finds its way into the bicarbonate. The carbonic acid enters by the opening k, and on the opening of the valve d, passes into the reservoir B; from thence it goes through f, and through



the apparatus A; it then passes into the reservoir C, and finally into the ammonia tower D, and from thence into the air. In the apparatus A are a number of divisions



connected with one another. The opening *v* serves for the continuous entrance of the alkali.—E. G. C.

*On the Production of Cyanides and Ferrocyanides from Trimethylamine by the method of Ortlieb and Müller.*  
Ed. Willm. Chem. Zeit. 50, 897.

THE well-known process for obtaining pure potassium carbonate from the chloride by means of carbonate of trimethylamine is carried on by the Joint Stock Company, of Croix. The author describes (in the Bull. Soc. Chim. 41, 449) the further successful adaptation at these works by Ortlieb and Müller, of the reactions first observed by Wurtz, which occur when trimethylamine, or either of the three methylamines, is passed through a red-hot tube. The amine is broken up into hydrocyanic acid, ammonium cyanide, and an inflammable carburetted gas. This is accomplished by evaporating the solution in small and constantly fed heating tubes, and passing the resulting vapour through red-hot retorts arranged similarly to gas retorts, thence into a condenser, and lastly into absorbing chambers, where the final separation takes place. These latter consist firstly of a series of vessels containing sulphuric acid, diluted sufficiently to prevent the crystallisation of the ammonium sulphate, which is formed by decomposition of the ammonium cyanide, when the gases are passed through the acid. Secondly there is a similar series, containing caustic alkali or milk of lime, to absorb the double quantity of hydrocyanic acid (*i.e.*, that originally present *plus* that resulting from the decomposed cyanide). Thus concentrated cyanide solutions are obtained, and the inflammable gas quite freed from ammonia and hydrocyanic acid, may be stored for lighting purposes. To produce ferrocyanides, a known quantity of pure ferrous hydroxide is added to the alkaline absorbent. This oxide is prepared by precipitating ferrous chloride with milk of lime, collecting and washing in a filter-press, and preserving the press-cakes from contact with the air until required for use. The solution of the solid hydroxide proceeds in propor-

tion as cyanides are formed in the liquid. After once filtering a ferrocyanide results equal in purity to that obtained by the ordinary method after recrystallising thrice.—W. G. M.

*The Recovery of Sulphurous Acid from Furnace Gases.*  
E. Häusch and M. Schreeder. Chem. Zeit. 48, 857.

THE authors describe a process with plant by the use of which sulphurous acid may be economically recovered, cold water being used as an absorbent. From the solution the sulphurous acid is again yielded on heating, the necessary heat being obtained by means of a system of water regenerators, which will be understood from the annexed cut. Fig. 1 is a ground plan, fig. 2 a section through AB, fig. 3 a somewhat enlarged cross section through EF. The furnace gases pass over *b* and *q* (described below), where they are cooled, and enter by *a* to the bottom of the tower *c*; after traversing this they are drawn off by means of a fan through the tube *d*. The tower *c* serves for the absorption of the sulphur dioxide contained in the gas; to this end, it is filled loosely with coke, shaped stones or wood lattice work, over which water is kept constantly flowing from above. The water containing the dissolved gas flows by the tube *e* (fig. 1) to the closed reservoir *f*, and thence through *g* to the lowest part of the warming apparatus *h*. This regenerator (fig. 3) consists of a number of large square flat compartments, each about 4c.m. high, made of sheets of lead 3m.m. thick, and piled one upon another, the alternate compartments being connected together by pipes at alternate ends. Thus the cold water, entering at the lowest compartment, traverses it and then rises by the pipe *i* to the third division; after passing through this it rises to the fifth, seventh, ninth, and so on. Meanwhile the hot water from a later stage of the process enters the top of the regenerator at *l* (fig. 1), passes through the uppermost compartment, sinks to that next but one below it, and so on to the second division from the bottom. The hot water thus gives up part of its heat to the cold solution in the intermediate compartments, and thus assists in the respective circulations of the two systems; it will be seen that the directions of the two currents are at right angles to one another. The sheet lead is supported by means of bearers of strip lead placed between each pair of plates, and in the direction of the current of water. A little heat is lost here, the amount varying with the time of contact, the size of the compartments and the velocity of flow; thus, with 10 to 12 minutes' contact, and 4 to 5 c.m. divisions, the temperature of the cold water will rise from 15° to 85°, that of the hot water sinking from 95° to 25° (loss = 10°). The warmed solution passes from *h* by *n* and *o* to the arrangement *b*, which consists of a series of closed leaden pans placed in, or forming the roof of, the channel through which the heated furnace gases pass to the absorption tower; here the solution becomes further heated to about 97° to 98°, and begins to yield the dissolved gas, which is carried by the pipe *p* to the main *u*. The compartment *g*, which is quite distinct from *b*, but similarly constructed, serves to cool the furnace gases still further by means of a constant current of cold water circulating through it. The hot solution is now conducted by the tube *r* to the top of the circular tower *s*, 4 to 5m. high and 2m. diameter. This tower contains a vertical lead-coated iron rotating shaft carrying at intervals of 0.25m. horizontal lead protected iron discs of 1.5m. diameter. Midway between each pair of discs is fastened to the wall of the tower a ring of similar material, with a central aperture of 1.0m. diameter. The hot solution falls upon the top rotating disc, and is thrown thence by centrifugal force onto the ring beneath, thence it falls upon the second disc, whence it falls upon the next ring, and so on. Meanwhile, the rarefaction of the air in the upper part of the tower, induced by a Körting's ejector in the pipe *u*, or by a hard lead Root's blower, causes air to enter by the pipe *t* at the bottom of the tower. The combined action of the heat, air, and agitation causes the solution to give up nearly the whole of its gas, which is passed by the main *u* to the vitriol

chambers, or wherever required. The hot spent solution collects in a reservoir beneath the tower, and is raised by a centrifugal pump to the reservoir *c*, whence it flows through *h*, as described, passing afterwards by *m* to the drains. Some simpler arrangement than that of rotation in *s* may be used, *e.g.*, lead wire gauze at distances of 0.1m. between each layer. The rate of flow of air and water is to be regulated by valves and cocks, and controlled by gas analysis. A small experimental plant (treating 50cub. m. of water in 24 hours, with a gas-evolving tower only 1.2m. high) over 99.5 per cent. of the gas dissolved in the water (3 to 4 kilograms per cub. m.) was regained, the temperature of the water being 97° to 98°, yielding a gas containing 28 per cent SO<sub>2</sub>. On a larger scale, complete recovery might be obtained at a lower temperature. With a flow of 2cub. m. per minute of water, containing 3 kilos SO<sub>2</sub> per cub. m., 432ctr. of chamber acid could be produced per diem, and the cost

Regenerating apparatus ( <i>h</i> )	30,000	"
Gas-evolving tower ( <i>s</i> )	3,000	"
Lead chambers of 1,000cub. m. capacity	80,000	"
with Gay-Lussac	15,000	"
Concentration, and various	211,000	Marks.

(1 Mark = 1 Shilling.)

—W. G. M.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, CEMENTS.

*Cement Containing Magnesia.* Dingl. Polyt. Journ. 252, 135.

ACCORDING to L. Erdmenger, of Misburg, magnesia calcined at a low temperature possesses the property, when added in certain quantities to Portland cement,

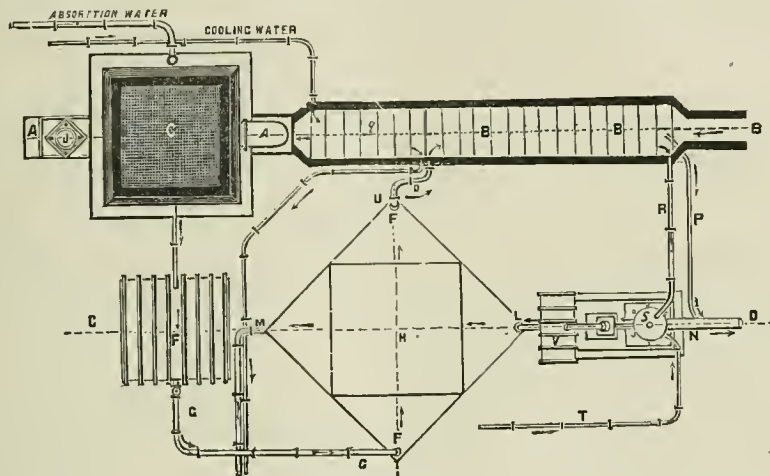


FIG. 1.

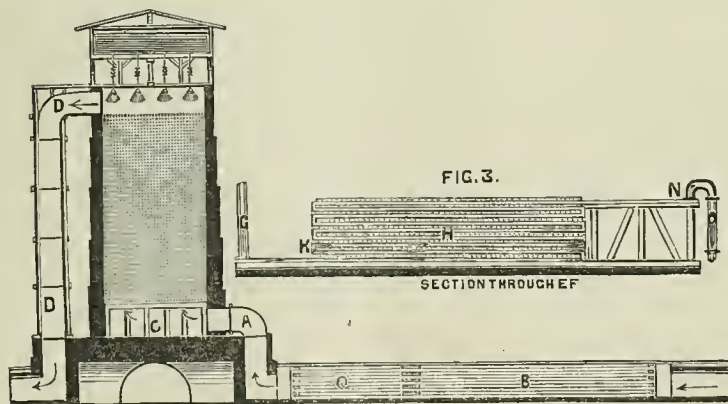


FIG. 2.

for raising the 2,880cub. m. of water, 20m. high, could not exceed 20pf. per lctr. of acid. Since the consumption of saltpetre is very small, and there is no other outlay for roasting pyrites, it is calculated that the total cost of producing chamber acid by this means would not exceed 50pf. per lctr. The cost of plant for a daily production of 400 to 500ctr. of chamber acid, working with furnace gases of one per cent SO<sub>2</sub>, is estimated at:

Engine for fan	12,000	Marks.
Steam boilers	12,000	"
Steam pump	9,000	"
Steam pump for hot water	5,000	"
Fan	7,500	"
Boiler-house and engine-room	12,500	"
Absorption tower ( <i>c</i> )	25,000	"
Plant for recovering heat of furnace gases	30,000	"

of rendering the latter more durable, and better able to withstand the action of the salts held in solution in sea water, etc.—E. G. C.

*Impregnation of Wood, etc., with Preservative Substances.* C. D. Abel, London. Eng. Pat. 5855, Dec. 24, 1883. Communication from abroad from J. A. Koch (Texas), and W. Hesse (Berlin).

THE object of the invention is to so treat wood and textile fabrics that they may be protected from atmospheric influences, and from the action of fire; also from decay when exposed to the action of sea water, etc. The materials to be preserved are first introduced into cold water, which is gradually heated to a temperature of from 170° to 248° F., to this there are added, sulphate of



iron, common salt or rock salt, and alum, in the proportions of 50 to 90 parts of sulphate of iron, 2 to 20 parts of rock salt, and 8 to 30 parts of common alum, to 50 gallons of water. The materials are boiled in this solution for some time, after which the bath is allowed to cool slowly, the material still remaining immersed therein. Further, instead of using above mentioned substances lime water may be employed as a preservative, and finally instead of using simply water as a bath in which, in the first instance, the material is immersed, a bath already prepared with chemical ingredients, or a spent bath may be used—A. J. K.

*Preserving and Hardening Wood.* Eng. Pat. No. 5519. Nov. 26, 1883. August Egestorff, Hull.

THE object of this invention is to effect improvements in means for preserving and hardening wood. The invention consists in applying to the wood certain reagents such as alkalis and phosphates, together with urine and certain salts. The wood is cut and boiled in a mixture consisting of—

Water	100lbs.
Urine	20lbs.
Burnt and unslaked lime 5lbs. and a little creosote tar.	

The proportion of ingredients and the time of boiling vary with the dimensions and character of the wood.  
—A. J. K.

*Treating Timber with Preservative Fluids.* S. B. Boulton, London. Eng. Pat. 5723, Dec. 13, 1883.

THE patentee claims the following:—(1) The process of treating wood with antiseptic substances wherein the wood is first treated with any of the substances referred to in his Eng. Pat. 62, 1882, and is then subjected to the action of a heated oily or bituminous body as described in his Eng. Pat. 1954, of 1879. (2) The process of treating wood first with a solution of metallic salts, and then subjecting it to processes described in Eng. Pat. 1954, of 1879. (3) The process wherein the wood is first treated with basic substances as acridine, lencoline, quinoline, etc., or with heavier or less volatile tar acids, and is then treated according to Eng. Pat. 1954, of 1879.  
—A. J. K.

*Dessicating Wood.* C. D. Abel, London. Eng. Pat. 5841, Dec. 22, 1883. Communicated from abroad by J. A. Koch (Texas), and William Hesse (Berlin).

THIS patent consists of a process for drying wood by imbedding it in dessicating substances, as "chloride of sodium or of calcium," also of mixing with such dessicators, some antiseptic substance, as carbolic acid or alum, whereby the wood is the better preserved.—A. J. K.

## X.—METALLURGY, MINING, Etc.

*Steel Smelting in Acid and Basic Crucibles.* By Albano Brand, Chem. Centr. 15, 407.

THE results of widely extended experiments are—  
*A in Acid Crucibles.*

1. In coke-clay crucibles and in graphite crucibles a considerable amount of carbon and silicon is taken up by the steel in the process.
2. In pure-clay crucibles there is a large gain of silicon with a corresponding loss of carbon by the steel.
3. In all three crucibles the molten product absorbs sulphur from the walls of the crucible.
4. Manganese promotes the coherence of the steel.
5. Indication on experimental foundation, how the pernicious influence of the crucible walls on the product may be avoided or diminished by choice and treatment of the substance of the crucible as well as by judicious management of the smelting process.

*B in basic crucibles.*

6. The author describes the preparation of magnesia crucibles for the smelting of larger masses at a very high temperature.

7. The author describes experiments for the discovery of a method for smelting large quantities of chemically pure iron for scientific purposes. Hereby iron and steel containing only one or two of the injurious (as P, Si, S, etc.), or of the advantageous admixtures (as W, Cr, Mn, etc.), can be produced and studied without the properties being obscured by the presence of others.—F. L. T.

*Coating Metal Plates with Tin, Etc.* E. Morewood, Llanelly, Carmarthenshire. Eng. Pat. 5938, December 31, 1883.

THE object of this invention is to so operate by mechanical means and appliances upon the sheets of metal emerging from the bath in which they are coated as to dispense with "the wash-pot or brushing over," and to yield covered sheets free from "scuff" and imperfections, and with a better finish than is otherwise given. This is accomplished by causing the sheets as they emerge from the bath to be guided by rollers, covered with molten grease so as to avoid exposure to the air, between "washing rollers" kept covered with molten metal. The washing rollers do not bear on the sheet, but are so closely adjusted as to wash off the scum and refuse on its surface. On leaving the washing rollers the plate, still passing through a bath of grease to prevent exposure, is passed through "finishing rollers" to remove the excess of metal, and finally through a pair of "improving rollers" which bear against each other by means of springs and thus lightly nipping the sheet between them impart a smooth surface and finish to the metallic covered sheet. The whole invention is covered by ten claims, which include the different processes in rotation, and the various details of the apparatus used.  
—C. C. H.

*Treating Ores to Purify and Prepare them for Smelting.* W. R. Lake, London. Communicated by C. Cherry, Senior, Chicago. Eng. Pat. 5788, December 18, 1883.

IN order to prepare certain kinds of ores for smelting and for the elimination of sulphur, phosphorus, arsenic, antimony, and oxygen, the process and apparatus described in this specification have been devised. A vertical retort is set in the interior of a chamber provided with a furnace, and communicates at its lower part with a tight gas chamber by means of a perforated bottom. A central flue is provided within the retort communicating by cross pipes formed in the top of the retort with the flues circulating round the exterior, such central flue being in communication with a chimney. The base of the retort is provided with a perforated pipe for the admission of a current of air, gas, or steam; and its upper portion is provided with a cap the only exit from which is through a safety valve capable of being loaded to any desired pressure. Chambers containing super-heating pipes and boxes are also provided, so that steam, gas, or air, admitted during different stages of the process into the retort, are super-heated by heat from the furnace. The process is conducted in three stages:

(1) No air is permitted to enter the charged retort, the exit valve adjusted so as to allow free escape for air or gases driven off from the ore and the temperature of the whole raised to about 700° F. Water of crystallisation is driven off, acid formations broken up, occluded gases and volatile substances expelled, and certain metallic oxides reduced. Fluxes, such as carbon, sodium chloride, or chloride of lime, may be used to break down silicious materials.

(2) The escape valve from the retort is then set to maintain a pressure of not less than 30lbs. per square inch in the interior, the temperature raised to 1000° F. and superheated steam admitted. Chemical decomposition at once occurs resulting in the formation of sulphuretted, phosphuretted, antimonetted, and arseniuretted hydrogen, &c., which are expelled and condensed.

(3) Steam is shut off, the exit valve regulated to maintain a pressure not exceeding 25lbs. per square inch, and superheated air admitted; the remaining combinations of sulphur, phosphorus, etc., not acted on in the second stage are now eliminated, and those bodies capable of

reoxidation are reoxidized. The ore is then ready for smelting. The whole invention is covered by 14 claims which set forth the novelty of construction of various parts of the apparatus, and that part of the process whereby ores are roasted in closed retorts in a current of gas, air, or steam, superheated, and at a pressure of not less than 30lbs.—C. C. H.

*Improvements in the Manufacture and Production of Oxide of Zinc.* Astley Paston Price, 47, Lincoln's Inn Fields, Middlesex. Eng. Pat. 5473. November 21, 1883.

THE oxide is produced by the combustion and oxidation of metallic zinc, contained in the gases from blast or other furnaces in which the metal is undergoing reduction, and is collected in suitably constructed chambers.—W. G. M.

## XL.—FATS, OILS, AND SOAP MANUFACTURE.

*Sand Soap.* By C. Roth, Seifensieder Zeitung, No. 21, 1884.

UNDER the name of sand soap a new detergent of peculiar appearance and composition, of English origin, has been recently placed upon the market. It is greyish white in colour, heavy as stone, and rough and sandy to the touch; on rubbing the hands with it particles of sand adhere to the skin. It is in fact an intimate mixture of fine sand with ordinary soap. In order to ascertain its composition, the writer has subjected it to analysis and finds it to contain 70 per cent. of fine sand; the saponified fat was palm oil. Experiments showed it to have no disagreeable or damaging effect on the hands when used as a detergent, and proved it moreover to be an excellent cleansing soap; especially suited for the use of workmen engaged in rough and dirty avocations. The author has made various experiments with a view to ascertain the maximum amount of sand which a soap of this kind can take up, and finds that 100 parts of soap can take up 200 parts of sand, but it is not advisable to add the full quantity as the mixture will no longer lather properly. To make a good "sand soap" the author recommends the following method:—100lbs. of cocoa-nut oil are saponified with about 200lbs. of lye at 20° B. The soap is then hardened by the addition of about 5lbs. salt dissolved in water to a density of 15° Beaumé, with addition of 6lbs. to 8lbs. soda ash. The soap is now covered up and the foam allowed to subside. After standing five to six hours the froth is skimmed and the soap is run off into the coolers. Whilst this process has been going on the sand has been dried and sifted, and the soap now being thoroughly crutched the sand is sifted over it until 100lbs. to 150lbs. have been added. The crutching must be continued until the mass is perfectly cooled. The soap is very firm and hard, and must be cut as soon as cooled. To perfume the mixture add of essential oil of lavender, thyme, and coriander 100 grammes each.—H. J.

*Transparent Glycerine Soap.* Seifensieder Zeitung [23], 1884.

THE manufacture of transparent glycerine soap does not present any especial difficulty; there are nevertheless points which it is essential to observe. To produce first-class soap of this kind good materials are indispensable, and the proper proportions must be strictly adhered to. Tallow and stearine are the most useful hard fats, palm oil imparts to the soap its lathering qualities and castor oil gives transparency. Great transparency is attained by the use of spirit, and of sugar water, both of which bodies assist the normal saponification. The lye must be pure, and clear as water; its proportion should not exceed 20 per cent. of lye per pound of fatty matter, for an excess of alkali would make the soap too detergent, and a considerable excess of unsaponified fat would make it too weak and greasy. In one case the soap injures the skin in use, in the other the soap would soon lose its agreeable smell and finally become rancid. Even an

excess of glycerine is to be avoided, as it renders the soap less transparent, and also too soft. No filling whatever except sugar solution is permissible. The following proportions are recommended:—

### WHITE ALABASTER SOAP.

13 lbs. stearin	18 lbs. 38° lye
22 " palm oil	26 lbs. 96 per cent. alcohol
13 " glycerine	

The stearin and palm oil are to be heated to 65°, saponified with the lye, the alcohol added and when the combination, which takes place at once is complete, the glycerine is put in. When clear, the kettle is covered and the contents are allowed to stand at 45° R. The soap is run into the moulds and perfumed with:—

120 grammes bergamotte oil	25 grammes neroli oil
30 " geranium	30 " citron "

As this is a white soap, no colour is added:—

### TRANSPARENT GLYCERINE SOAP.

20 lbs. tallow	20 lbs. spirit
12 " palm oil	20 " glycerine
8 " castor "	5 " sugar dissolved in
20 " 38° lye	5 " water

Process as above. Perfume with—oil of bergamotte 250 grammes, citron 90 grammes, lavender 20 grammes, neroli 30 grammes, rosemary 5 grammes, and a few drops of otto of roses, dissolved in one pound of 96° alcohol colour with saffron substitute.—H. J.

*Vegetable-oil Residues.* Seifensieder Zeitung [23], 1884.

THESE residues, consisting of partly saponified oils together with various impurities separated out of the original oil in the process of refining now find profitable employment in various branches of the soap boiling industry, as a cheap material for mixing purposes.—H. J.

*Professor Hermann's Apparatus for the Valuation of Lubricants.* Berg und Hüttenm. Zeitung.

THIS apparatus consists of a perfectly cylindrical shaft about 10cm. in length supported in two journals carried by a branched support which turns on a hinge, so that the shaft can be adjusted at an angle of 5 to 10 degrees to the horizon; the upper end of the shaft carries a handle, whereby it can be rotated. To use the apparatus, a bent piece of the same metal etc., as that to which the lubricant is to be applied, is placed astride of the shaft, and suitably weighted, and the number of turns of the handle requisite to cause it to slide along the shaft for a given distance with various lubrication, are noted. The greater the distance travelled for a given number of revolutions of the shaft, the more efficient the lubrication.—H. J.

*Notes on the Indian Oil Trade.* Seifensieder Zeitung. No. 23, 1884.

THE import of vegetable oils into India was a very important one by comparison with that of previous years, especially with regard to Ceylon cocoa-nut oil. The import of vegetable oils which, in the year 1881 and 1882, only amounted to 96,541 gallons rose in 1883, to 534,003 gallons. Mineral oils, principally American kerosine, show an increase as follows:—

	Galls.	Rupees
1878-79	3,775,974	2,717,112
1879-80	7,888,247	4,819,079
1880-81	10,060,026	4,870,580
1881-82	9,883,049	5,040,326
1882-83	21,059,665	9,293,123

Thus, the increase in quantity during the five years equals 45.8 per cent., while the total value has only increased 38.4 per cent. Kerosine oils came mostly from America direct, the other descriptions from England. The petroleum industry in the North of India and in Burmah is now attaining considerable proportions and bids fair to render the country independent of the American supply.—H. J.



*On Vegetable Wax.* Max Buchner (Chem. Centrallbl 1884, 257). Repert. anal. chem. 9, 137.

UNDER this denomination, formerly restricted to carnauba, palm-, myrtle-, or Japan-wax, comes a kind of fat exported to Austria from the East Indies, which is undoubtedly the fat of a species of *Bassia*. Fat is obtained in large quantities in the East Indies, and in parts of Africa, from the seeds of this genus of plants, and is known by different names according to the species of *Bassia* from which it is derived, such as galam-, bambui-, bambara-, or Shea-butter, from *Bassia parkii*; by other names when obtained from *Bassia butyracea*; *Bassia* oil or Illipe oil, when from the East Indian mahwah-, or butter-tree; and similar fats are yielded by *Bassia latifolia* and *Bassia longifolia*. Some of these fats closely resemble palm oil and taste like butter, while they may be preserved for a long time unaltered; others soon become rancid and liable to decomposition. A specimen of one of these fats examined by the author possessed about the same consistency as butter, a rancid taste, and greenish colour. The fat was not homogeneous, and imbedded in the green amorphous mass were numerous crystalline aggregations, the character of which could be distinguished by the aid of the micro-polariscope. The melting point of the green amorphous fat was 33.6°, and that of the crystals, 55.6°. The specific gravity was 0.9474. This fat was little soluble in alcohol, boiling absolute alcohol only taking up 1.68 per cent., and cold alcohol only 0.83 per cent. The composition of *Bassia* fat is very variable: O. Henry found stearin to predominate. Pelonze and Bourdet discovered chiefly olein. Buff detected no palmitic acid, while Thomsen and Wood discovered a new acid, identical with stearic acid and with a melting point of 70°. According to Valentia, the fat of *Bassia longifolia* yields 63.49 per cent. of oleic acid, and 36.51 per cent. of a fatty acid, of 62° melting point, and essentially the same as palmitic acid. The vegetable wax analysed by the author yielded 80 per cent. of a fatty acid, melting at 60°, and consisting principally of palmitic acid.—E. G. C.

*On the Adulterations of Volatile Oils.* H. W. Langbeek. Repert. Anal. Chem. 12, 177.

THE author has met with a sample of citronella oil adulterated with 15 per cent. of turpentine; a so-called Algerian oil of geranium, consisting of a mixture of French and Indian oil of geranium; and a sample of Ylang-Ylang oil (*oleum unguis odoratissimae*) consisting of a mixture of orange-flower oil, partially oxidised bitter almond oil, oil of cardamoms, oil of pimento, and oil of cinnamon. Among the various methods employed for the detection of adulterations in volatile oils, may be mentioned fractional distillation of the suspected oil, and examination of the condensation-products. This method is, however, frequently inapplicable, owing to the smallness of the quantity of oil at disposal. Another method consists in allowing a drop of the suspected oil to evaporate spontaneously on a piece of filtering paper, and recognising by the odour the presence of any admixture: but this process is not applicable to oils containing oxygen. The polariscope method gives unreliable results, as has been shown by Flückiger, the polarising properties of an oil varying with the time it has been kept, and depending on differences in the soil, temperature, time of gathering in of the plants, etc. Heppe's reagent, nitroprusside of copper, serves to detect the adulteration of oxygenated oils with those free from oxygen, but not oxidised or unoxidised oils among themselves. The method proposed by Zeller and Heppe, and worked out by Dragendorff, based on the greater or less solubility of the pure oils, and of those mixed with oil of turpentine, oil of copaiba, etc., in alcohol, yields better results, but reliance can only be placed on it when one works with oils in a fresh condition. The author has found that oils from plants belonging to the family of the Labiate dissolve large quantities of salicylic acid, and those from the family of the Umbellifere, with few exceptions smaller quantities, while scarcely any of this acid is soluble in oils from the Conifere, Dicteraceae, and

Cassiae—the results of a considerable number of observations are given in a table appended to the paper. The method used was as follows:—A number of flat-bottomed reagent-glasses, about two inches long, and 5-16ths inch in diameter, were each charged with 0.05 grms. of salicylic acid, and then accurately weighed. Various oils, known to be pure, were next added, drop by drop, to the separate portions of salicylic acid. Each glass was then shaken, oil being added until a clear solution was obtained; the increments of weight now gave the comparative solubilities. The same oil was next tested, after being mixed with five per cent. and ten per cent. respectively of oil of turpentine. The ethereal oils, as brought into the market, almost always contain small quantities of water, to the presence of which is due the formation of hydrogen peroxide in larger or smaller quantities, according to the length of time that the oils have been preserved. The author estimates the quantity of peroxide approximately, and also indirectly, the age of the oil, by the aid of potassium iodide and starch paper. The oil to be examined is shaken with its own bulk of water, and, after complete separation, a drop of the latter is placed upon the test paper: if the oil be fresh or recently rectified, the paper remains white, but old oil strikes a more or less intense colour, which, with similar oils, of which the age is known, allows, with a little practice, of extremely accurate conclusions being drawn.—E. G. C.

## XII.—PAINTS, VARNISHES, AND RESINS.

*Manufacture of White Lead.* G. Hand Smith, Chelsea. Eng. Pat. 5862. December 26, 1883.

THE object of this invention is the production of white lead by a method and process which, while securing the conditions which exist in the manufacture of this substance by the Dutch process, dispenses with the use of tan, bark, or decomposing vegetable matter, and affords a better control over its production, as well as being more certain and rapid in its operation. The metallic lead is exposed in a rectangular vessel, in a suitable form, to the action of air charged with carbonic acid and acetic acid vapour, as well as aqueous vapour. This corroding chamber is enclosed in an outer vessel, which may be supplied with hot air, or a water-jacket heated by suitable means, so as to regulate the temperature of the interior vessel. The air supplied, charged with carbonic acid gas, is passed through a saturating vessel containing diaphragms of a textile fabric wetted with an aqueous solution of acetic acid, and is supplied at a slight pressure, being permitted to expand in the corroding chamber, and thus by depositing aqueous vapour ensure more rapid chemical action. A regular temperature of 120° F. to 140° F. is found most advantageous for the reactions. The proportion of air and carbonic acid gas found to work well is 20 per cent. of CO<sub>2</sub> to 80 per cent. of air, and a pressure equal to about six inches of water before entering and expanding in the corroding chamber has been found suitable. The aqueous solution of acetic acid found to work best in the saturation is that containing about 0.75 per cent. of acid. This proportion admits of variation, and in some cases nitric acid may be also used with advantage, or a tray of concentrated acid or acids, as described, may be placed on the floor of the corroding chamber. The claims are (1): The process as above described for the manufacture of white lead by acid vapours, air, and water vapour, supplied under pressure, and allowed to expand in a corroding chamber; (2) the use in the manufacture of white lead of the apparatus, as described and specified.—C. C. H.

## XIII.—TANNING, LEATHER, GLUE AND SIZE.

*On the Examination of Tanning Extracts.* F. Simand (Der Gerber. 1883, 211). Repert. Anal. Chem. 9, 136.

THE author employs the Löwenthal-Neubauer method, which he prefers to any other. Some commercial ex-

tracts, analysed by him, have yielded the following results, from which it will be seen that the solutions prepared with hot water gave higher figures than those made with cold water :—

EXTRACT FROM	Dissolved in cold water, percentage of Tannin.	Dissolved in hot water, percentage of Tannin.	Difference for 100 parts of Extract.	Corresponds to 100 parts of Tannin (calculated.)	REMARKS.
Quebracho Wood .....	70.09	73.08	2.99	1.09	The Commercial Extract, dried before Analysis.
Valonia .....	68.59	70.44	1.85	2.62	Prepared by himself, dried before Analysis.
Oak-wood .....	15.09	15.47	0.38	2.45	18 B., prepared by himself.
Fir .....	13.72	14.31	0.59	4.13	32 B. } Commercial Ex-
Oak-bark .....	23.72	24.37	0.65	2.67	32 B. } tracts.
Chestnut Wood .....	22.68	23.52	0.84	3.57	31 B. }
Sumach .....	10.75	13.38	2.63	19.66	31 B. }

—E. G. C.

#### XIV.—AGRICULTURE, MANURES, Etc.

*Method for Estimating Phosphoric Acid in Superphosphates.* E. Aubin. Compt. rend. 98, 1591.

COMMERCIAL superphosphates frequently contain a certain amount of phosphate, which has escaped the action of sulphuric acid. Nenbauer and Fresenius recommend the use of ammonium citrate to determine the quantity of decomposed and undecomposed phosphate. The treatment of superphosphates with this reagent may be effected in different ways. In the absence of magnesia the product is ground up direct with the reagent, whilst in its presence it is necessary to use water. In the latter case, the superphosphate is exhausted repeatedly with small quantities of water, and decanted on a small filter, or digested with a large volume of water after grinding. In both cases the portion insoluble in water is filtered off and digested with ammonium citrate. These different modes of treatment do not give the same results. The author has investigated this subject, and attributes the differences to the following causes: (1) The degree of acidity of the superphosphates; (2) the grinding of the superphosphates during the operation of sampling; (3) the presence of large quantities of magnesia in the superphosphates. The results of the analyses of fifteen samples of superphosphates are given to confirm the author's views as to the causes of these differences, and it is recommended to extract all soluble substances before treating the superphosphate with ammonium citrate.

—D. B.

*Use of Decorticated Cotton Cake.* By DeLa Tréhonnois. Journ. de l'agric. 768 of 1883.

THE substitution of decorticated cotton cake for an equal quantity of copra cake in the feeding of four milch cows caused a considerable improvement in the yield of milk.

—H. J.

*Experiments with Nitrate of Soda.* By M. Klowitter-Wulka. Allgm. Ztg. No. 89, 1883.

PLOTS of barley were manured with various quantities of nitrate of soda; the lowest results were yielded when one half of the nitrate was sown with the seed, and the rest added subsequently as top-dressing. The yield on the other plots improved with the increased quantity of manure applied.—H. J.

*Influence of Krugite on the percentage of Starch in Potatoes.* By M. Kette-Jassen, Wochenschr. de P. rek. Gesellsch. No. 4, 1884.

THE use of Kainit as a potato manure is known to cause a diminution of the percentage of starch in the tubers. This effect has been attributed to the high percentage of

chlorides in kainit, and it appeared, therefore, probable that the use of krugit, a potash salt composed chiefly of sulphate of potash and gypsum, with very little chloride would obviate this disadvantage. In every instance,

however, the use of a manure containing krugit caused a serious diminution in the percentage of starch.—H. J.

*The Manufacture of Bone Meal.* By Prof. Koenig. Chemiker Zeitung. No. 29 of 1884.

UNTIL recently, bones intended for the production of bone meal were previously boiled or steamed for the purpose of extracting the fat; this method is now superseded by the benzene process, of which the author describes three modifications, employing the solvent respectively in form of vapour at ordinary pressure (Leuner's process) or aided by heat and pressure; (Seltam's process), or in vacuo with subsequent heating and pressure; (Richter's process). The analysis of a number of samples of bone meal gave results favourable to the benzene process, and the author points out that these latter, whilst effecting a more complete and profitable extraction of a substance (fat) valueless from a manurial point of view, has a further advantage over the steaming process in that it exerts no injurious effect upon the gelatine of the bones.—H. J.

*The Use of Nitrate of Soda in Beetroot Culture.* Braunschweiger Landw. Zeitung. No. 50, 1883, and No. 5, 1884.

A NUMBER of fresh experiments have been made in various districts for the purpose of ascertaining the value of nitrate of soda as a manure for the sugar beet. The results indicate a decided improvement in weight of crop without diminution of the percentage of sugar in the roots, resulting from a cautious use of nitrate of soda together with phosphates, the proportion proved to give the best results being 2 parts of nitrate to every 3 parts phosphoric acid. The use of sulphate of ammonia (equal money value) in place of nitrate of soda did not in general improve the results.—H. J.

*The Manuring of Barley.* By H. Wäterling. Braunschw. Landw. Zeitung. No. 6, 1884.

THE results of a series of experiments upon the yield of barley grown respectively without manure, and with various artificial fertilisers showed that the use of phosphates with a little nitrate of soda, as also that of nitrate of soda alone, caused no increase, and even in some cases a positive diminution in the yield of grain. The best results were obtained by the use of 1 centner superphosphate, with from 1 to 2 centners nitrate of soda per morgen.—H. J.



*Occurrence of Nitrates in the Vegetable Kingdom.*  
Berthelot. Compt. rend. 98, 1506.

To detect the presence of potassium nitrate in plants the author employed the following method: 200 to 300 grms. of plant are extracted with water, the solution is evaporated on a water bath, and the residue taken up with a mixture of alcohol and water. The solution is again evaporated to expel the alcohol, and the residue tested for nitrates according to Schlösing's method. Possibly the method recently described by Arnaud and Padé may be more successfully applied to detect the quantity of nitrates. This method is based on the conversion of nitric acid or nitrates into cinchonamine nitrate by treatment with cinchonamine hydrochloride. The stalks of plants contain by far the largest proportion of nitrates as illustrated by the following analyses, which refer to the first periods of vegetation of the relative plants:—

Amarantus candatus:	(stalk	0.0204g.	KNO <sub>3</sub>
Weight of a dried section of the plant:	roots	0.0039g.	"
	0.610g. (leaves	0.0024g.	"
marantus with red leaves:	(stalk	0.0054g.	"
Weight of a dried section of the plant:	roots	0.0011g.	"
	0.518g. (leaves	0.00036g.	"
Borrago officinalis:	(stalk	0.0270g.	"
Weight of a dried section of the plant:	roots	0.0026g.	"
	1.1195g. (leaves	0.0058g.	"
Delphinium:	(stalk	0.1600g.	"
Calculated on 1,000 parts of the dried	roots	0.0140g.	"
plant	leaves	0.0000g.	"
	blossom	0.0000g.	"
Medicago sativa:	(stalk	0.00018g.	"
A dried section of the plant	roots	0.00170g.	"
Triticum sativum:	(stalk	0.00023g.	"
A dried section of the plant	leaves	0.00031g.	"
Avena sativa:	(stalk	0.0032g.	"
Weight of a dried section of the plant:	roots	0.0009g.	"
	2.80g. (leaves	0.0011g.	"

The author has determined the amount of potassium nitrate in a large number of plants, with the following results:—

steam. The raw sugar is sifted, so as to get rid of all balls and lumps, and is then placed in a battery of suitably shaped vessels, similar to the diffusion battery used in beetroot sugar factories. A saturated solution of pure sugar is then forced, or sucked through the mass of crystals, and successively passed through the different vessels in either an upward or downward direction. The aqueous solution in its progress carries with it the coloured impure syrup, leaving behind pure crystals, both from the sugar operated upon and that contained in the saturated lixiviating solution. The claim is made for the method and process, and for the purpose substantially as described.—C. C. H.

## XVI.—BREWING, WINES, SPIRITS, Etc.

*On Brewing.* C. Lintner. Chem. Zeit. 1884. 68, 435, 450, 505, and 751.

IN the middle and west of Europe (Austria with the exception of the south of Hungary, in the middle and south of Germany, France and England), the common long-eared barley is cultivated, the most important breweries producing malt from this variety. It has been demonstrated by Schulze and others that the weight of the grain is no criterion as to the amount of dry matter, starch, proteids and ash contained in barley. The average weight of 1 hectolitre of barley varies from 64 to 65 kilos, 66 to 68 kilos is a large weight, whilst 70 to 72 kilos is considered an extreme case. The grain of barley should possess a pale straw yellow colour, grains exhibiting different colours are objectionable, especially if they have yellow or brown tops. Barley grains should be bellied and plump in structure, and be surrounded by a thin skin, their embryo should be intact and of greenish yellow colour, the presence of damaged grains being conducive to the formation of fungi in the malt. The impurities in barley amount to from 0.35 to 4 per cent. In order to

Name of Plant.		Potassium Nitrate in 1000 parts	
		Dried Plant.	Fresh Plant.
Hypnum triquetrum (Moss)	—	0.055	0.050
Equisetum Telmateia (Equisetaceæ)	Stalk.	0.360	0.066
Pteris aquilina (Filices)	"	0.300	0.053
Scirpus lacustris (Cyperaceæ)	"	0.019	0.012
Juncus conglomeratus (Juncaceæ)	"	0.180	0.065
Asparagus officinalis (Liliaceæ)	Bulbs.	0.300	0.044
Scilla nutans (Liliaceæ)	Stalk.	0.077	0.024
Dactylis glomerata (Graminaceæ)	"	0.110	0.021
Triticum sativum (Graminaceæ)	"	27.800	4.400
" eight days later	"	11.200	2.700
Avena sativa (Graminaceæ)	"	9.500	1.030
" eight days later	"	17.600	2.800
Pinus sylvestris (Conifereæ)	Young sprouts.	0.210	0.049
Prunus domestica (Rosaceæ)	"	0.120	0.026
Pyrus communis (Rosaceæ)	"	0.150	0.043
Papaver Rhoeas (Papaveraceæ)	Stalk.	31.600	1.600
Chelidonium majus (Papaveraceæ)	"	2.200	0.240
Solanum tuberosum (Solanaceæ)	"	15.400	1.060
Bryonia dioica (Cucurbitaceæ)	"	33.300	2.100
Plantago lanceolata (Plantaginææ)	"	0.770	0.150
Lychnis dioica (Caryophyllaceæ)	"	1.900	0.230
Galium aparine (Rubiaceæ)	"	0.100	0.012
Cherophyllum temulum (Umbellifereæ)	"	0.180	0.020
Euphorbia Cyparissias (Euphorbiaceæ)	"	Traces.	—
Geranium robertianum (Geraniaceæ)	"	7.000	0.780
Senecio vulgaris (Compositæ)	"	0.490	0.071
Tanacetum vulgare (Compositæ)	"	0.750	0.076
Urtica dioica (Urticaceæ)	"	12.600	1.800
Lamium album (Labiaceæ)	"	0.190	0.033
Reseda lutea (Resedaceæ)	"	5.900	0.740
Brassica alba (Crucifereæ)	"	2.800	0.480
Rumex acetosa (Polygonææ)	"	0.380	0.042
Ranunculus acris (Ranunculaceæ)	Leaves.	0.150	0.018
Trifolium pratense (Leguminosæ)	Stalks.	Traces.	—
	"	"	—

—D. B.

## XV.—SUGAR, GUMS, STARCHES, ETC.

*Improvements in Refining the Coloured Raw Sugar of Commerce so as to Produce White Crystalline Sugar.*  
C. Stellen, Vienna. Eng. Pat. 5746. June 9, 1884.

THE object of this invention is to free raw crystalline sugar from all impure syrup without the employment of

determine the germinating power of barley, 200 grains (or preferably more) are allowed to germinate between sheets of moist blotting paper and two glass plates, and from the development of the grains the percentage of sprouted grains is calculated. After some practice trustworthy results are obtained by this method. Under

favourable conditions 96 to 98 per cent. of the grain of barley should sprout, 90 per cent. is considered unfavourable, and below 90 per cent. very unsatisfactory. Stainer's apparatus for estimating the germinating power of barley consists of a white glazed stoneware basin covered with a layer of sand. An earthenware plate, having 100 indentations, each forming a receptacle for one barley grain, is placed on the layer of sand, and the apparatus is covered with a bell jar. Fresh air is introduced through the bottom of the dish and the germinating plate, the escape being effected through an aperture in the top of the bell jar. Thus pure air is constantly contained in the germinating space. The grain is steeped in water and placed on the germinating plate, previously moistened, together with the layer of sand on which the plate rests. According to Aubrey, barley contains on an average 1.682 per cent. of nitrogen, corresponding with 10.513 of proteids, the average percentage of phosphoric acid being 1.056. The action of water on barley during the process of steeping has been the subject of study by various investigators. Experiments made by Mills and Pettingrew have shown that water used for the purpose of steeping barley contains at least two albuminoid bodies, one of which is precipitated by metaphosphoric acid in the cold, the other on boiling. The former can be wholly kept back within the grain by a solution of gypsum, and probably, but with less efficiency, by a solution of calcium carbonate. The effect of all lime salts is to keep back nitrogenous matter within the grain. Burton water abstracts more nitrogen, or at least as much as either gypsum or chalk water. The special esteem in which this water is held is thought to be due to the nitrates contained in it, which, as is well known, promote the germination of the seed. To work with as little loss as possible, it is recommended that a water containing 0.1 per cent. gypsum be used, to which a small amount of calcium nitrate may be advantageously added. If, however, just sufficient water to saturate the grain be used, soft water may then be employed without experiencing any loss. Ullrich found that potash and magnesia are removed in the greatest amount by hard water, whilst distilled water extracts a large proportion of soda and phosphoric acid. Hard waters do not only prevent the removal of lime, but give up large quantities of their lime to the grain. Heut has investigated the influence of distilled water on the composition of various barleys, and his results confirm the conclusions which Ullrich has arrived at. Kjeldahl's investigations on diastase have shown that for the conversion of starch into sugar a temperature ranging between 54° and 63° gives the best results. It has been demonstrated also that during its conversion into green malt the diastatic action of barley is increased very considerably, whilst during the operation of kiln drying it is reduced by about one half, hence this operation is one of the most important points in brewing. To obtain a light coloured beer, the temperature at which the malt is dried, should not exceed 33 to 50°. It is not, however, profitable to dry at too low a temperature. For the production of medium fine beers (Vienna beers) the drying is effected at a temperature of from 75 to 87.5°, the length of time to be given to the drying being 16 to 24 hours. At the commencement the temperature shows 20 to 30° in the upper part, and 56 to 68° in the lower part of the heap. If the drying is completed in 16 hours, the maximum temperature is reached in 5 or 6 hours; but if the drying requires 24 hours, the highest temperature is arrived at in from 8 to 10 hours. The malt contains about 40 per cent. of water when placed on the drying floors. It loses the greater proportion of moisture whilst on the upper floor, so that on its arrival at the bottom floor it contains only 3 to 4 per cent. of moisture. The defects in the present malting processes are said to be due mainly to the want of uniformity of temperature in the germinating heaps, and the difference in the amount of moisture which exists in the various layers. The gradual discharge of carbonic anhydride and the defective admission of oxygen has a marked effect on the malt. These imperfections are said to be removed by the so-called "pneumatic malting process," in which the barley, after germination, is thrown on a plate having fine perforations; the air underneath the

plate is then exhausted. Thus the heat is withdrawn, and the carbonic anhydride removed in a regular stream, so that the germinating grain is situated in a bath of pure air, having always the same temperature, and containing the same amount of moisture. To effect this, the air is previously purified and heated to the necessary temperature. For the successful storing of malt during the summer, it is necessary to protect it from moisture and from the action of insects. According to Schulze young malt always produces an opalescent wort, whilst the same malt, after seven weeks' storage, gives a perfectly clear wort, the albuminous substances which are the cause of this opalescence being gradually converted into an insoluble form. The production of the worts may be divided into four processes:—(1) Crushing the malt, (2) Steeping and mashing, (3) Boiling the worts and adding the hops. And (4) Cooling the worts.

If malt is ground instead of being crushed, the yield of wort extract is largely increased. Coarsely crushed malt gave 57 per cent. of wort-extract, whereas finely crushed malt produced 63.54 per cent. When a decoction is employed, it is best to stir up the malt with cold water (8°), and it is found that a slow increase of temperature from the commencement gives the most favourable results. The length of time to be given to the mashing is from 4 to 5 hours. The mashing at the Klein-Schweehacht brewery in Vienna is conducted in the following manner:—The malt is stirred up with cold water, and the temperature raised to 35° by the addition of hot water. The temperature of the first mash is 55°, and of the second mash 65°. The extract is then mashed a third time at a temperature of 72°. The length of time for boiling the first thick mash is 20 minutes, for the second mash 30 minutes, and for the so-called "Lauter-maisch" 40 minutes. In the infusion process the mash is not boiled, the requisite temperature being obtained by the addition of hot water or by the use of "dry" steam. The crushed malt may be added to the proper amount of mash water previously heated to from 75 to 85°, and the mash allowed to stand for one or more hours, or the malt may be added to the cold mash water, and the temperature raised by steam heat to 65–70°, after which the infusion is left at rest for one or more hours. The latter process gives the best results. The proportion of malt to water is 1:2. Schulze has recently examined the best conditions for the production of malt extract and maltose in beer mash, and concludes that the infusion process is no better than the decoction process. The quantity of hops to be added to the wort depends on the quality of the beer to be brewed. German beer requires from 0.15 to 0.85 kilo. per hectolitre, namely, 0.15 to 0.40 kilo. for young beer, 0.3 to 0.6 kilo. for Lager beer, and 0.5 to 0.85 for export beer. For bottom fermentation the hot wort is cooled to 5 to 6°, and for top fermentation to 12 to 20°. For this purpose the wort is poured out into large shallow cisterns or coolers. As the time for cooling should not exceed 8 hours, it often becomes necessary to cool the wort by artificial means. The top fermentation process is employed for beers which are intended for immediate consumption or contain a large amount of extract such as English beer, which is prepared from highly dried malt, with a considerable addition of hops, and consequently contains a larger proportion of substances capable of arresting fermentation. Bottom fermentation is used for worts, containing a small proportion of sugar or extract. Both processes are divided into the primary and secondary fermentation. The former represents that period of fermentation in which in a comparatively short time a considerable amount of the sugar is decomposed, and the strongest appearances indicative of fermentation are noticeable, such as evolution of carbonic anhydride, copious separation of yeast and visible increase of temperature. This period requires from 2 to 22 days for its completion according to circumstances. When the primary fermentation is finished, the secondary fermentation commences and goes on slowly for weeks and months, until it ceases for want of sugar, and the product is no longer beer. The bottom fermentation of beer worts is carried on in open circular or oval oak vats, larch being used occasionally. The vats have a capacity of 20 to 35



hectolitres, and are placed under ground. The temperature of the fermenting cellar is kept at about 5 or 6°, whilst the fermentation of the worts is conducted at a temperature approaching but not exceeding 9°. The heat is kept down by ice or cold water, or by the so-called "ice swimmers." The yeast is added to the cooled worts in a moist or dry state. 1000 litres of worts require about 46 litres of yeast pulp. In the case of lightly hopped beers (running beer or young beer) the primary fermentation process is finished in 8 to 10 days, whilst with heavily hopped beers (Lager beer) from 12 to 14 days are required. The fermentation is completed when the density of the fermenting liquor ceases to be reduced perceptibly. The beer is then run off into casks, having previously removed the thin brown scum which floats on the surface, and contains the resins of the hops. This, if allowed to sink, would impart a peculiar bitter flavour to the beer. In the casks the secondary fermentation process proceeds slowly. The temperature at which the fermentation is started in the top fermentation process varies from 10 to 20°. The amount of yeast used is the same as that employed in bottom fermentation. The wort is fermented either in open vats or in barrels. The fermentation is continued for a period of from 48 to 60 hours, and is then checked by drawing off the liquor into barrels. In these the fermentation still goes on. The yeast as it rises works out at the bung, and constitutes bakers' yeast. The secondary fermentation is finished when, instead of yeast, a white scum works out at the bung. The clear beer is then racked off into barrels and preserved for use. The so-called "white beers" are top fermentation beers. For their preparation a mixture of barley and wheat malt is used, the latter gives the beer its characteristic taste. In conclusion, it is stated that until recently top fermentation-beer only was produced in England, but owing to the rapid progress which has been made in the production of artificial ice, the brewing of German or bottom fermentation-beer has been commenced in England.—D. B.

*Non-alcoholic Beverages.* Agatha C. L. Weigel. Brighton. Eng. Pat. 5506. Nov. 24, 1884.

THE object of this invention is the manufacture from grapes, or other fruits, of beverages containing all the constituents of good genuine fermented wine with the sole exception of the alcohol. The fermented juice of the grapes is submitted to a process of fractional distillation in a vacuum pan—the volatile ethers coming off first, and then the alcohol—the former are returned to the juice, and the latter, which is pure, is sold for medicinal or chemical purposes. The liquor, which has been entirely freed from alcohol, is now drawn off through worms kept cold by running water and through filters from which all air is rigorously excluded into an airtight reservoir, where it is kept at a temperature just above its freezing-point. It is at this point that the ethers are returned to it, and there is also added a portion of condensed unfermented graped or fruit juice, specially prepared for the purpose. The liquor thus prepared can be bottled as a "still" beverage, or as an aerated or sparkling beverage by being impregnated with carbonic acid gas. The whole operation and apparatus are so arranged that the juice from the moment it enters the vacuum pan until it is bottled can be entirely excluded from contact with air.—A. J. K.

*Improvements in and Relating to the Treatment and Utilisation, Chiefly as Cattle Food, of the Residuary Products of the Distillation of Spirits, and Apparatus therefor.* W. R. Lake. Eng. Pat. 1485. Jan. 15, 1884. Communicated from abroad by Georg. Baumert, Ph.D., Halle-on-the-Saale.

THE object of this invention is to transform into a dry state all the solid substances contained in the residuary liquors from the distillation of ardent spirits, and to so treat the said solid substances that they may be stored away, for use as cattle food, for any length of time—the general custom being to mix the liquors straight away with some suitable fodder material; others prepare a

mixture which will only serve for immediate use. The process consists essentially of a separation, first, of all of the solid from the liquid constituents of the residuary liquors. The former are then merely dried, and the latter concentrated by evaporation, mixed with some suitable fodder and with the above-mentioned solid constituents, and the mass finally dried. The details of the process, and the special plant required, are fully described.—A. J. K.

*Treatment of Hops in Brewing.* F. C. Glaser. Eng. Pat. 5811. Dec. 19, 1884. Communicated by Albert Kempe, Moscow.

THE author of this invention claims thereby to make a saving of about 20 per cent. in the amount of hops used in the ordinary process of manufacturing beer. He points out that the addition of hops to beer serves two distinct purposes. In the first place the wort is boiled for  $3\frac{1}{2}$  hours with hops in an open pan, whereby the albumen in the wort is precipitated by the tannic acid of the hops and by the resinous constituents of the lupulin. The aroma which should have been imparted by the volatile essential oils of these hops is lost, owing to the oils having volatilised—hence it becomes necessary to make a fresh addition of hops to the cold wort to impart the desired aroma—and the other properties of this last addition of hops are thus made no use of. By the present process, instead of making this second addition of hops, an extract of hops is prepared by means described in the specification, and a portion of this extract is added to the cold wort.

—A. J. K.

## XVII.—FOOD PRESERVING, MEATS, Etc.

*Use of Boric Acid for Preserving Food.* J. Forster. Chem. Centr. Bl. 1884, 675.

THE author concludes that the admission of boric acid as an addition to food results in injury to the digestive organs, inasmuch as its action tends materially to increase the proportion of solid matter separated in the intestinal canal. Its use as a food preservative is not as beneficial as has been hitherto assumed.—D. B.

## XVIII.—SANITARY CHEMISTRY, DISINFECTANTS.

*Application of Domestic Utensils Containing Lead.* H. Fleck. Chem. Centr. Bl. 1884, 676.

FROM a series of experiments made with enamelled pots, the author concludes that their behaviour to boiling vinegar varies considerably. He found that under certain conditions enamels containing lead were not affected by vinegar. An enamel from which 0.041 gram. lead per litre capacity had been dissolved, was prepared by the same manufacturer from the same raw materials, the only difference being that it was burnt more strongly. This enamel was found to resist entirely the action of boiling vinegar.—D. B.

*Antiseptic Properties of Potassium Bichromate.* Lan-jorrois. Chem. Centr. Bl. 1884, 676.

TO test the antiseptic action of potassium bichromate, 500grms. of urine were treated with five grms. of this salt, and exposed for eight months. During this period the urine remained perfectly clear and transparent, without smell, and free from all signs of putrescence. Blood when treated in a similar manner gave the same results. Cow's milk, treated with one per cent. potassium bichromate, remained unaltered during three summer months. Owing to its poisonous action this salt cannot be used as a preservative for food.—D. B.

*Analyses of Certain Waste Waters.* Prof. Koenig Landw. Zeitg. f. Westfalen. No. 12, 1884.

THE author gives detailed analyses of a number of samples of waste waters discharged from sugar works, paper mills, bleach works, pit heaps, etc. Amongst the

solid constituents of the pit heap waters a high per centage of chloride of sodium is noticeable in some instances, together with much free sulphuric acid from the oxidation of iron pyrites. Four samples of town waste water (without water-closet contamination) contained respectively in milligrammes per litre :

	Sample 1.	Sample 2.	Sample 3.	Sample 4
Suspended matter } organic	307.2	289.0	—	387.0
} inorganic	246.4	202.2	9.2	—
Dissolved matter } organic	2407.9	2180.4	458.2	3010.0
} inorganic	884.8	898.0	1628.0	1532.0
Lime .....	—	—	—	117.0
Magnesia .....	—	—	—	28.0
Potash .....	—	—	—	81.0
Soda .....	—	—	—	181.0
Sulphuric acid .....	109.8	110.0	137.5	12.0
Chlorine .....	135.8	143.2	610.0	181.0
Sulphuretted hydrogen .....	2.6	1.8	6.8	traces
Ammonia .....	65.1	39.9	7.5	—
Oxygen requisite for oxidation	120.4	109.0	22.9	152.0

—H. J.

*On the Hygienic Significance and Detection of Carbonic Oxide.* A. P. Fokker. Chem. Cent. Blatt. 20, 380.

THE author speaks of the work of Gruber (Chem. Cent. Blatt. 81, 709—82, 809), criticizes Fodor's method for determining carbonic oxide (Chem. Cent. Blatt. 80, 669), and advances the statement that for the correct determination of small quantities of carbonic oxide it is not to be relied on. (1) It is not certain in passing air through palladium chloride solution that the whole of the carbonic oxide is left behind. If we take two instead of one absorption apparatus filled with the palladium chloride solution, we find a reduction has taken place in the second solution before the first is quite reduced. It therefore shows the possibility of traces of the gas passing through with the air, and thus impairing the correctness of the observation. The author has often been unable to find small quantities of carbonic acid by this method. (2) Before passing the air through, the blood must be largely diluted, and the air to be tested must already have been shaken up with diluted blood. This proceeding must falsify the experiment. (3) The method of Fodor labours under the disadvantage that in drawing air through, the diluted blood scums over, whereby the palladium chloride solution is decomposed, especially as, according to Gruber, to obtain the reaction with small quantities of the gas, the passage of the air must be continued for three or four hours. The author then gives the following modification of the process :—1 to 2ccm. of the blood which has to be tested for carbonic oxide is put into a small, rather deep beaker glass clamped between three bent brass wires. The upper end of the latter supports a watch-glass, containing a little palladium chloride solution, while the lower ends are soldered to a round brass plate. The apparatus is then put into a porcelain vessel filled with water, and covered with a narrow bell-jar. Two-thirds of the air are then sucked out of the bell-jar by means of a stiff indiarubber tube, inserted near the beaker. The water then rises, and the beaker, kept upright by the brass plate, swims on the surface of the water. By means of a lamp the water is brought to the boiling point, the blood coagulates, and the palladium chloride solution is reduced by the disengaged carbonic oxide. If the blood contains only traces of the gas, the reduction is not complete, and the apparatus must be allowed to stand for 24 hours. The water on cooling will, to a certain extent, rarify the air in the bell-jar, and thus facilitate the separation of the carbonic oxide from the blood. A reduction of the palladium chloride shows the presence of carbonic oxide. Free ammonia gives, with palladium chloride, a yellow amorphous compound, while that produced by carbonic oxide is a brilliant black metallic mirror undergoing no change. The same appearance is produced by sulphuretted hydrogen, which, however, is seldom found in fresh blood. The delicacy of this estimation is almost unlimited, and it is possible to detect carbonic oxide in a single drop of blood, which cannot be done by using any other method.—G. H. B.

## XIX.—PAPER, PASTEBOARD, Etc.

### *Impermeable Packing Paper.* Papier Zeitung.

A MIXTURE of caoutchouc and paraffine, or of caoutchouc and Japanese wax gives a glutinous paste impervious to water and free from smell. This composition spread between two sheets of brown paper makes a cheap flexible, perfectly waterproof packing material.—H. J.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

### *Purification of Methylalcohol.* J. Regnaud and Villejean. Compt. rend. 99, 82.

LIEBEN obtained pure methylalcohol by treating methyl oxalic ether with a base. The methyl alcohol obtained by the action of calcium hydrate on methyl oxalic ether always contains a certain quantity of a substance yielding iodoform when treated with iodine and alkaline hydrates. Possibly this substance is acetone, which is always present in the commercially "pure" methyl alcohol, and is probably retained by the oxalic ether. It might also be a trace of ethyl alcohol. The authors propose to treat the regenerated alcohol with a large amount of iodine (about one-tenth its weight), and render the mixture alkaline with soda. On careful distillation pure methyl alcohol is said to be obtained, which when rectified over lime has a sp. gr. of 0.810 at 15°. The object of this treatment is to convert all foreign matter, which may be present in the alcohol after the oxalic acid treatment, into iodoform, and decompose the latter into iodide and alkaline formate.

—D. B.

### *Detection of Alcohol in the Volatile Oils.* Th. Salzer. Report. Anal. Chem. 9, 138.

FUCHSIN dissolves in alcohol, but is insoluble in the volatile oils. This circumstance has for many years been made use of for the detection of alcohol in ethereal oils. The author does not appear to have been acquainted with this well-known reaction.—E. G. C.

### *Quantitative Estimation of Morphia in Opium.* Von Perger (Journ. Pr. Chem. 1884, 97). Report. Anal. Chem. 9, 137.

AMONG the various methods for the quantitative estimation of morphia in opium, the author particularly recommends that of E. Merck, as being the most useful. The process is as follows :—15grms. of finely cut opium are boiled with 100grms. of 45 per cent. spirit of wine, the extract so obtained being then separated from the residue by filtration. The residue is once again treated with 100grms. of alcohol of the same strength, and the filtered extracts are mixed with 8grms. of crystallised soda, and evaporated without being stirred. The residue of evaporation is now washed with 60grms. of cold water, then with 45grms. of 90 per cent. alcohol, and finally put on a filter. The crystalline mass remaining on the filter is dried between sheets of blotting-paper, dissolved in a mixture of 15grms. of acetic acid (consisting of one part acetic acid, of sp. gr. 1.06, and eight parts water) with 15grms. of distilled water, and filtered through the filter on which the residue was collected; the filtrate is precipitated by ammonia, and the precipitate received on a tared filter, after 12 hours' standing, dried and weighed.

—E. G. C.

### *Otto of Roses and its Adulterations,* by E. Moride. Seifensieder Ztg. No. 21, 1884.

THE production of the essential oil of roses is chiefly carried on in Turkey. The trade in the article is nearly all centralised at Kestanlik. The method of producing it is as follows :—10 kilos. of dry roses of the strongly scented muscat variety are placed in a still of peculiar shape, together with 70 kilos. of water, and the distillate collected by a simple condenser; this gives about 20 kilos.



of rose water, which is redistilled, and the drops of otto of roses floating upon the surface of the product are collected. Otto of roses is soluble in warm alcohol, and is a mixture of a fluid, nitrogenous essential oil, and a crystallisable, non-nitrogenous stearoptene, which latter is most abundant in samples from colder regions, which consequently congeal more readily. Turkish oil congeals at from  $11^{\circ}$  to  $16^{\circ}$ . It is generally admitted that the otto of roses is not adulterated by the makers, but its high price occasions a great deal of fraudulent mixture in second hands, and the methods of analysis relied on to detect this leave much to be desired. Besides spermaceti and animal oils geranium, rosewood (*convolvulus scoparius*), and sandal wood oils, even copaiba and alcohol have been used for this purpose.—H. J.

## XXII.—GENERAL ANALYTICAL CHEMISTRY.

*A New Method for the Detection of Arsenic.* H. Hager (Pharm. Centralhalle 1884, 265). *Repert. Anal. Chem.* 12, 183.

A DROP of the hydrochloric acid solution of arsenic is placed on a clean brass plate, and evaporated over the very small flame of a petroleum lamp. The result is a dark patch possessing the colour of potassium permanganate, but with weaker solutions of arsenic a reddish tint only is obtained, while with very dilute solutions a faintly-coloured border to the patch only is noticed. The drop must be heated gradually at first, but the temperature must at no time be sufficient to bring about the volatilisation of ammonium salts. The arsenical solution must not contain either free ammonia, free sulphuric acid, free nitric acid, or a nitrate, any one of which will interfere with the reaction. The author gives detailed instructions for applying his test to the detection of arsenic in sulphuric, hydrochloric, phosphoric, and acetic acids; also in carbonates, bismuth subnitrate, etc.—E. G. C.

*Detection of Acetic Acid in Formic Acid.* By H. Beckurts (Pharm. Centralh. 1884, 54). *Repert. Anal. Chem.* 8, 123.

ACCORDING to the second edition of the German Pharmacopœia, if one gramme of formic acid be heated for ten minutes with five grammes of water and one gramme of oxide of mercury, the fluid filtered off from the reduced mercury should not possess an acid reaction, the equation being:  $\text{HCOOH} + \text{HgO} = \text{Hg} + \text{CO}_2 + \text{H}_2\text{O}$ . Any acetic acid present would remain unaltered, and would impart to the filtrate an acid reaction. The author remarks that one gramme of oxide of mercury is too little, 1.17 grm. of the oxide being theoretically necessary for one gramme of acid, and he has found, on testing commercial samples of mercuric oxide, notable quantities of water and of calomel. The latter amounted, in two samples examined, to 0.72 and 1.07 per cent., respectively, while the former varied, in three samples, from 2.25 to nearly three per cent. These impurities are objectionable, as they diminish the actual weight of mercuric oxide taken, and the presence of calomel, moreover, hinders the reduction of the formate of mercury. An excess of oxide of mercury will not interfere with the detection of acetic acid, as acetate of mercury has an acid reaction.—E. G. C.

*Apparatus for Drying and Washing Gases.* By Joh. Walter. *Chem. Cent. Blatt* 20, 369.

THIS consists of a Woulff's bottle with two necks, Fig. 1. A conducting tube C is passed through one of the necks, while a wide tube A, the lower end of which is narrowed, passes through the other. This latter tube can be closed at the top, near which it has a small outlet tube D. A funnel tube B is introduced so that its lower end protrudes through the narrow end at a. In order to prepare the apparatus for use, the funnel-tube is first inserted, so that the funnel is just outside the top of A, while the lower end still shows outside a. While held in this position the space between the two tubes is filled

with glass pellets up to h; this being accomplished the funnel-tube is drawn down till it rests on the glass packing, the remainder of A being afterwards nearly filled with the glass pellets. When the apparatus is in use,

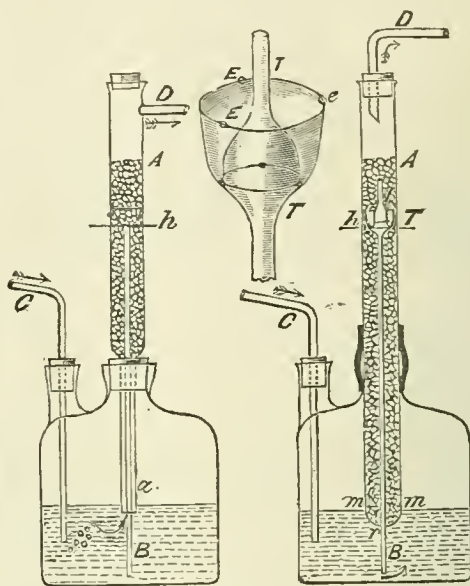


FIG. 1.

the gas to be manipulated is conducted by the tube C into the apparatus, entering tube A between B and a, passing up through the glass pellets, and out at D, any liquid which may have been carried up by the stream of gas falling back into the funnel, and so back to the bottom of the apparatus. A more effective apparatus is that shown in Fig. 2, in which the wide glass tube A reaches down to the liquid. The gas passes through four openings in the tube, up through the glass pellets, and out at D. The opening r allows the passage of the funnel-tube B. The funnel T has three or four glass pellets fused upon its upper rim. It is recommended that in this funnel a second and smaller one should be inverted, having also three glass pellets fused to its rim. These pieces of apparatus are suitable only when the stream of gas is regular.—G. H. B.

*Regulator for Fractional Distillation in Vacuo.* By L. Godefroy. *Chemisches Centralblatt* 15, 386.

THE apparatus consists chiefly of two wide vertical tubes, A and B (Fig. 1), joined below by a narrow tube. The tube A has a cock R above, and over that a funnel. The tube B ends above in a narrower tube N, over which an indiarubber tube may be pushed; it is provided below with a three-way cock R<sub>1</sub>, and in the side, at a distance of 10–12 mm., are fused two tubes which soon turn upwards. These tubes have a diameter of about two millimetres. One of them ends in the bulb G, which again terminates in the tube M, over which an indiarubber tube can be pushed. The other tube enters the bulb G, and then bends downwards. The tube A starts full of mercury, the other tube B contains more or less mercury according to the vacuum to be produced. To use the apparatus, it is connected with the suction-pump and the distillation apparatus, the former at M, the latter at N. The pump is now started, the mercury sinking in A and rising in B. The surface first reaching the opening of the tube C, and then that of E. From this moment it remains stationary, for the air entering at N not only prevents the mercury from rising higher than E, but endeavours to press its surface down. The opening E hereby becomes free, and an air bubble rises in EF; the

mercury blown up with it running back through DC. In this way a constant pressure is obtained equal to the difference of level in A and B. In the apparatus of fig. 1 the limb EF occasionally becomes somewhat obstructed. The pressure in the apparatus increases by

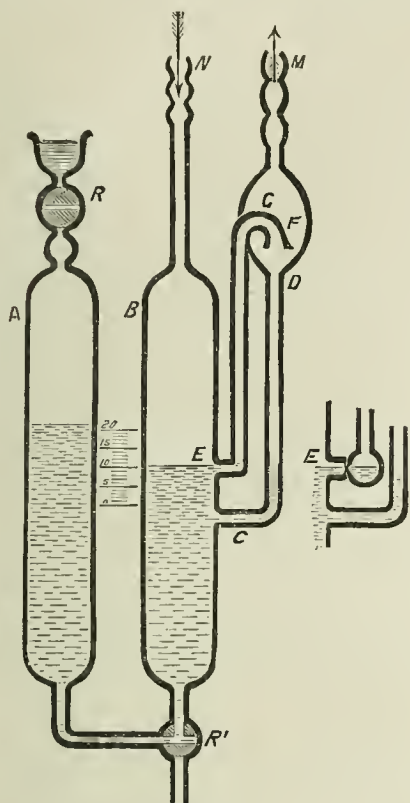


FIG. 1.

a fraction, without, however, interrupting the regularity of the boiling. This little inconvenience is obviated by the modification of the opening E in the figure. The apparatus may be employed for very different pressures.

vacuum regulator by the two cocks R and R'. Finally the tubes  $t_3$  and  $t'_3$  are provided with two cocks  $r$  and  $r'$ , by which communication can be made with the air. If R and R' are opened, and  $r$  and  $r'$  closed, the vacuum is produced in the apparatus. When G is so placed that the condenser is only connected with the bottle A, the distillate collects in the latter. By turning G round, the distillate can be conducted into B. On closing the cock R and opening  $r'$ , the bottle A fills with air, and can be removed.—F. L. T.

#### *A New Reagent for Sodium, Ammonium, and Lithium.* H. Hager. Pharm. Centr. 25, 291.

THE reagent in question is potassio-stannous chloride prepared by dissolving five parts crystallised stannous chloride in 10 parts water, and adding a solution of potash of 1.145 sp. gr. in quantity sufficient to produce an almost clear solution. After an hour's standing, five parts potash and 15 parts water are added. The mixture is then filtered, and the colourless solution kept in bottles fitted with indiarubber stoppers. The reaction consists in the formation of a white precipitate. The solution to be examined should not be very acid. The presence of earthy or metallic salts and of boric acid interferes with the reaction. In testing caustic potash for soda, and potassium carbonate for sodium carbonate, it is preferable to saturate the alkali partially with hydrochloric acid, whereby the delicacy of the reaction is increased.—D. B.

#### THE PREVENTION OF THE POLLUTION OF RIVERS BILL.

The Council begs to call the attention of members to the subjoined abstract of a "Rivers Pollution Bill," prepared and brought in by Mr. Hastings, Earl Percy, and Colonel Walrond, 13th Nov., 1884, and invites its consideration by the sections at the earliest opportunity.

1. This Act may be cited for all purposes as the Rivers Pollution Prevention Act, 1885.

2. Every person who—

(a.) Puts or causes to be put or to fall, or permits to be put or to fall, or to be carried into any stream, so as, either singly or in combination with other similar acts of the same or any other person, to interfere with its due flow, or alter its bed, or pollute its waters, any solid matter from any

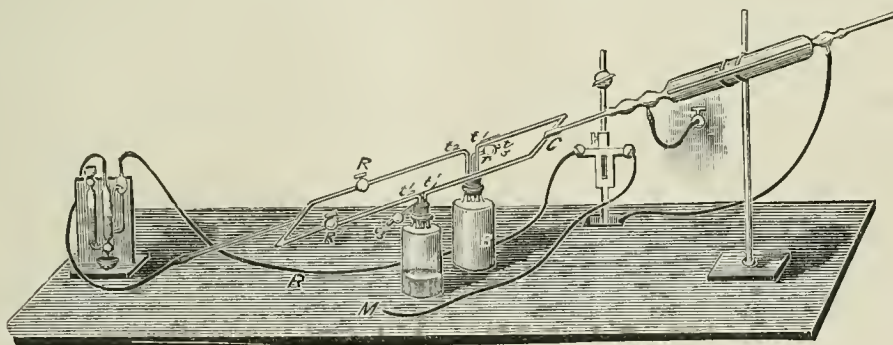


FIG. 2.

To regulate it, the limb B is almost entirely emptied of mercury, and the pump and distillation apparatus are connected. As soon as the level in B has become stationary, as much mercury is run into A as produces the required pressure. In fig. 2 the whole apparatus for distilling is depicted. The two wide-necked bottles A and B are closed with three holed indiarubber corks, through which pass three glass tubes. The tubes  $t_1$  and  $t'_1$  communicate by the three-way cock G with the condenser. The tubes  $t_2$  and  $t'_2$  are connected with the

manufactory, manufacturing process, mine, quarry, or place, where any trade business, or occupation is carried on, or any rubbish, cinders, refuse, or waste, or any solid matter, putrid, poisonous, offensive, or injurious to animal life;  
(b.) Puts, or causes to fall or flow, or permits to be put, or to fall or flow, or to be carried into any stream, any liquid or solid sewage matter;  
(c.) Puts, or causes to fall or flow, or permits to be put or to fall or flow, or to be carried into any



stream, any liquid containing matter poisonous, noxious, polluting, or injurious to animal life from any manufactory, manufacturing process, mine, or place where any trade, business, or occupation is carried on ;

shall be guilty of an offence against this Act.

In proving any offence against this Act, evidence may be given of repeated acts, which, taken together, may cause such offence, although each act, if taken by itself, may not be sufficient for that purpose.

It shall be sufficient to constitute an offence against this Act to prove that the person charged with the offence has done some act or acts prohibited by this Act, although the pollution complained of may not have been entirely caused by him.

Provided always, that it shall be no defence to any offence against this Act to prove that after any offence against the Act has been committed, the water of the stream is less polluted than is defined by the standards of purity herein-after mentioned.

3. The county court having jurisdiction in the place where any offence against this Act is committed may, by summary order, require any person to abstain from the commission of such offence, and may insert in any order such conditions as to time, or mode, or means of prevention of the pollution, or other action in the matter, and give such directions as to carrying the said order into effect, as it may think just.

The Court may direct any skilled persons to report as to the best mode or means to be adopted for preventing any pollution, and may make the adoption of such mode or means by the person who has committed such offence part of its order: Provided always, that the court shall not sanction the adoption of any mode or means of action for prevention of pollution that does not reduce the polluting matter to a less extent than is defined by the standards of purity herein-after mentioned.

Any person making default in complying with any requirements of any order of a county court made in pursuance of this section, shall pay to the person complaining, or to such other person as the court may direct such sum, not exceeding *fifty pounds a day* for every day during which he is in default as the court may order, and such penalty shall be enforced in the same manner as any debt adjudged to be due by the court; moreover, if any person in default persists in disobeying any requirement of any such order for a period of not less than *a month*, or such other period less than a month as may be prescribed by such order, the court may, in addition to any penalty it may impose, appoint any person or persons to carry into effect such order, and all expenses incurred by any such person or persons, and such amount as may be allowed by the county court shall be deemed to be a debt due from the person in default to the person or persons executing such order, and may be recovered accordingly in the county court.

4. If either party, in any proceedings before the county court under this Act, feel aggrieved by the decision of the court in point of law, or on the merits or in respect of the admission or rejection of any evidence, he may appeal from that decision to the High Court of Justice and to the Court of Appeal. The appeal shall be by motion, or, if from the judgment after trial in the county court, in the form of a special case, and such case shall be agreed upon by both parties or their solicitors, and if they cannot agree, shall be settled by the judge of the county court upon the application of the parties or their solicitors.

The court before whom such appeal is heard may draw any inferences from the facts stated in the case that a jury might draw from facts stated by witnesses.

Subject to the provisions of this section, all the enactments, rules, and orders relating to the proceedings in actions in county courts, and to enforcing judgments in county courts and appeals from decisions of county court judges, and to the condition of such appeals, and to the powers of the superior courts on such appeals, shall apply to all proceedings under this Act, and to an appeal from such action, in the same manner as if such action and appeal related to a matter within the ordinary jurisdiction of the court.

Any plaint entered in a county court under this Act may be removed into the High Court of Justice by leave of any judge of the said High Court, if it appears to the said judge desirable in the interests of justice that such case should be tried in the first instance in the High Court of Justice, and not in a county court, on such terms as to security for and payments of costs and such other terms (if any) as such judge may think fit.

5. It shall be the duty of and incumbent upon the sanitary authority of the district within which any offence against the Act is alleged to be committed, on the complaint either of any conservancy authority having jurisdiction over the stream in which such offence is alleged to be committed, or of any fishery board within the district of which such offence is alleged to be committed, or of any owner or occupier of land or a dwelling-house within the district of the sanitary authority, to institute proceedings to restrain the commission of such offence, and if the sanitary authority refuse to institute such proceedings, or having instituted the same, refuse or neglect to prosecute the same with due diligence, such conservancy authority or fishery board or owner or occupier may apply to the judge of the court in which such proceedings should be instituted or are pending for leave to institute proceedings in the name of, or to continue and prosecute and conduct in the name of, the sanitary authority, the proceedings instituted, and the court may permit the same proceedings to be so instituted or continued and prosecuted at the cost of the sanitary authority. All necessary and proper costs or expenses incurred by or on behalf of the applicant under and in accordance with the aforesaid provisions of this section and not recoverable from the offender shall be payable to the applicant by the sanitary authority, and all such payments, and all other proper expenses incurred by the sanitary authority under and in pursuance of the aforesaid provisions of this section, shall be deemed expenses properly incurred by such authority in the execution of the Public Health Act, 1875.

Proceedings may also be instituted in respect of any offence against this Act by any person aggrieved by the commission of such offence. Provided always, that the court in which such proceedings shall be brought may, on the application of the person against whom such proceedings are taken, order the person who institutes the same to give security for the costs of such proceedings, and, unless such security be given, may order such proceedings to be stayed.

6. If, in the opinion of the Local Government Board, after a local inquiry by a duly qualified inspector appointed for the purposes of this Act, it is desirable that the order of any court directing a person found guilty of an offence against the Act to use proper and sufficient modes or means, or certain prescribed modes or means, for the prevention of a pollution to any stream within a limited time should be modified by allowing an extension of time, or the adoption of some other mode or means, such person may, with the consent of the Local Government Board, apply to the court for an order allowing such extension of time, or the adoption of some other mode or means, and the court may, after hearing the evidence adduced in support of and against such application for such extension of time, or for the adoption of some other modes or means, modify any existing order accordingly. Provided that such other mode or means reduce the polluting matter below the standards of purity herein-after mentioned; and provided also that every order of the court modifying any existing order shall be subject to the same appeal as the existing order would be subject to; and that in no case shall a greater extension of time than *six months* be made by any court.

7. The powers given by this Act shall not be deemed to prejudice or affect any other rights or powers now existing or vested in any person or persons by Act of Parliament, law, or custom; and such right or power may be exercised in the same manner as if the Act had not passed, and nothing in this Act shall legalise any act or default which would, but for the Act, be deemed to be a nuisance or otherwise contrary to law, nor shall

apply to affect the lawful exercise of any rights of impounding or diverting water.

8. Any person who causes any polluting matter to fall or flow into any stream by any channel, drain, or other communication that has been made or opened since the fifteenth day of August one thousand eight hundred and seventy-six, shall incur a penalty not exceeding *fifty pounds*, and a further penalty not exceeding ten pounds a day for each day, dating from the date of the first conviction, such penalty to be recovered in manner provided by the Summary Jurisdiction Acts. In any proceedings under this section, the burden of proving that the channel, drain, or other communication by which the polluting matter passes or flows into any stream, was made or opened before the fifteenth day of August one thousand eight hundred and seventy-six, shall rest with the person causing the polluting matter to pass or flow into any stream.

9. In this Act, if not inconsistent with the context, the following terms have the meanings herein-after respectively assigned to them (that is to say) :—

“Person” includes any body of persons corporate or unincorporate.

“Court” means the county court of the district in which the pollution of any stream takes place.

“Stream” includes all rivers, whether tidal or otherwise, brooks, navigable canals, lakes and water-courses, except such parts of a tidal river as the Local Government Board may by order, published in the “London Gazette,” after local enquiry determine on sanitary grounds not to be part of a river. It shall also include so much of the sea as the Local Government Board may by like order, and on the like ground, declare to be part of a stream.

“Sanitary authority” means in the metropolis, as defined by “The Metropolis Management Act, 1855,” any local authority acting in the execution of “The Nuisances Removal Act for England, 1855,” and the Acts amending the same; elsewhere in England any urban or rural sanitary authority acting in the execution of “The Public Health Act, 1875.”

“Conservancy authority” means any body of conservators having, by Act of Parliament, jurisdiction over any river for navigation or other purposes.

“Fishery board” means a board of conservators appointed under “The Salmon Fishery Acts, 1861 to 1876,” or “The Freshwater Fisheries Acts, 1878 or 1884.”

“Standards of purity” shall mean—

(a.) Any liquid which has not been subjected to perfect rest in subsidence ponds of sufficient size for a period of at least six hours, or which, having been so subjected to subsidence, contains in suspension more than one part by weight of dry organic matter in one hundred thousand parts by weight of the liquid, or which, not having been so subjected to subsidence, contains, in suspension, more than three parts by weight of dry mineral matter, or one part by weight of dry organic matter in one hundred thousand parts by weight of the liquid.

(b.) Any liquid containing in solution more than two parts by weight of organic carbon, or  $\frac{3}{4}$  part by weight of organic nitrogen in one hundred thousand parts by weight.

(c.) Any liquid which shall exhibit by daylight a distinct colour when a stratum of it, one inch deep, is placed in a white porcelain or earthenware vessel.

(d.) Any liquid which contains, in solution, in one hundred thousand parts by weight, more than two parts by weight of any metal except calcium, magnesium, potassium, and sodium.

(e.) Any liquid which, in one hundred thousand parts by weight, contains, whether in solution or suspension, in chemical combination or otherwise, more than  $\frac{1}{10}$  part by weight of metallic arsenic.

(f.) Any liquid which, after acidification with sulphuric acid, contains, in one hundred thousand

parts by weight, more than one part by weight of free chlorine.

(g.) Any liquid which contains, in one hundred thousand parts by weight, more than one part by weight of sulphur, in the condition either of sulphuretted hydrogen or of a soluble sulphuret.

(h.) Any liquid possessing an acidity greater than that which is produced by adding two parts by weight of real muriatic acid to one thousand parts by weight of distilled water.

(i.) Any liquid possessing an alkalinity greater than that produced by adding one part by weight of dry caustic soda to one thousand parts by weight of distilled water.

(k.) Any liquid exhibiting a film of petroleum or hydrocarbon oil upon its surface, or containing, in suspension, in one hundred thousand parts, more than  $\frac{1}{10}$  part of such oil.

10. In the application of this Act to Scotland, the following provisions shall have effect :—

(1.) The expression “sanitary authority” shall mean and include the local authority in any parish or borough in Scotland acting under the Public Health (Scotland) Act, 1867.

(2.) The expression “London Gazette” shall mean Edinburgh Gazette.

(3.) The expression “The Public Health Act, 1875,” shall mean the Public Health (Scotland) Act, 1867, and any Acts amending the same.

(4.) This Act shall be read and construed as if for the expression “the Local Government Board” wherever it occurs therein “the Secretary of State” were substituted, and the expression “the Secretary of State” shall mean one of Her Majesty’s principal Secretaries of State.

(5.) The expression “the County Court” shall mean the sheriff of the county, and shall include the sheriff substitute, and the expression “plaint entered in a county court” shall mean petition or complaint presented in a sheriff’s court.

(6.) The expression “the High Court of Justice” shall mean the court of session in either division of the Inner House thereof.

(7.) All the jurisdiction, powers, and authorities necessary for the purpose of this Act are hereby conferred on sheriffs and their substitute.

(8.) The court of session may, on the application of the Lord Advocate on behalf of the Secretary of State, interpose their authority to any order made by the Secretary of State as to the costs incurred by him in relation to inquiries instituted by him under this Act, and as to the parties by whom such costs shall be borne, and may grant decree conform thereto, upon which execution and diligence may proceed in common form.

(9.) An inspector appointed for the purposes of this Act by the Secretary of State shall, for the purposes of any inquiry directed by the Secretary of State under this Act, be entitled by a summons, signed by him, to require the attendance of all persons he may think fit to call before him in regard to the matters of the inquiry, and to administer oaths to and examine upon oath all such persons, and to require and enforce the production upon oath of all documents, accounts and papers in anywise relating to such inquiry, and shall also have in relation to the inspection of places and matters required to be inspected similar powers to those which sanitary inspectors have under the Public Health (Scotland) Act, 1867.

11. In the application of this Act to Ireland the following provision shall have effect :—

(1.) The expression “sanitary authority” shall mean any urban or rural sanitary authority acting in the execution of “The Public Health (Ireland) Act, 1878 :—”

(2.) The expression “the Public Health Act, 1875,” shall mean the “Public Health (Ireland) Act, 1878 :—”

(3.) The expression “the Local Government Board”



shall mean the Local Government Board for Ireland :

- (4.) The expression "the county court" shall mean the civil bill court :
- (5.) The expression "plaint entered in a county court" shall mean Civil Bill process :
- (6.) The expression "the High Court of Justice" shall mean Her Majesty's High Court of Justice in Ireland, and the expression "Court of Appeal" shall mean Her Majesty's Court of Appeal in Ireland or any part thereof to which appeals may be brought from the decision of a civil bill court :
- (7.) The expression "the judge of the county court" shall mean the chairman of quarter sessions and judge of the civil bill court :
- (8.) The expression "the London Gazette" shall mean the Dublin Gazette :
- (9.) All the jurisdiction, powers, and authorities necessary for the purposes of this Act are hereby conferred upon the civil bill courts and superior courts and the judges of the same respectively.
- (10.) All penalties when recovered by or on behalf of, or at the instance of, or in any proceeding instituted by any sanitary authority, or any officer of such authority, shall be paid to such sanitary authority, and by the same applied in aid of their expenses under the Sanitary Acts; and, save as aforesaid, all such penalties shall be applied, and the penalties mentioned in section eight shall be recovered and applied in manner directed by "The Fines Act (Ireland), 1851," and any Act amending the same.

12. The Rivers Pollution Prevention Act, 1876, is hereby repealed.

## New Books.

**INORGANIC CHEMISTRY.** By EDWARD FRANKLAND, Ph.D., D.C.L., LL.D., F.R.S., Professor of Chemistry in the Normal School of Science, and FRANCIS R. JAPP, M.A., Ph.D., F.I.C., Assistant Professor of Chemistry in the Normal School of Science. With Illustrations. London: J. & A. Churchill, 11, New Burlington Street, 1884.

A LARGE 8vo. volume, bound in red cloth, with map of spectra as frontispiece, preface, table of contents, introduction (Chap. I) on "Matter & Force," and forty chapters of subject matter besides, the whole covering 783 pages. A complete alphabetical index closes the work, and this index covers twenty-one pages, the last page also including a small list of errata. The work is illustrated by some 51 woodcuts. The chapters are headed as follows, these headings giving a fair idea of the plan of the whole work:—Chap. I., matter and force; II., elements and compounds; III., chemical nomenclature; IV., laws of combination; V., the atomic theory; VI., molecular weights; VII., atomic weights; VIII., chemical notation, atomicity; IX., compound radicals; X., classification of elements; XI., relations between chemical composition and specific gravity, atomic volume; XII., chemical affinity; XIII., chemical homogeneity; XIV., isomerism, metamerism, polymerism, allotropy; XV., heat of chemical combination, thermo-chemistry; XVI., fusion and fusing-points; XVII., ebullition and boiling-points; XVIII., solution; XIX., diffusion; XX., crystallography; XXI., weights and measures. **NON-METALS;** XXII., monad elements; XXIII., dyad elements; XXIV., triad elements; XXV., tetrad elements; XXVI., pentad elements; XXVII., hexad elements; XXVIII., monad elements (halogen group); XXIX., tetrad elements (silicon group); XXX., pentad elements (phosphorus group). **METALS;** XXXI., distinguishing characteristics of the metallic ele-

ments; XXXII., monad elements (potassium group); XXXIII., dyad elements (barium group); XXXIV., dyad elements (cadmium group); XXXV., triad elements (gold group); XXXVI., tetrad elements; (aluminium group); XXXVII., metals of the rare earths; XXXVIII., tetrad elements (platinum group); XXXIX., tetrad elements (lead); XL., hexad elements (uranium group); XLI., hexad elements (chromium group).

**CHEMIKER-KALENDER.** 1855. Ein Hilfsbuch für Chemiker, Physiker, Mineralogen, Industrielle, Pharmaceuten, Hüttenmänner, u. s. w. von Dr. RUDOLF BIEDERMANN, Sechster Jahrgang. Mit einer Beilage. Berlin, 1885. Verlag von Julius Springer. Monbijouplatz, 3.

THIS well-known Taschenbuch has just appeared for 1885, and it is to be followed during this month (November), by Dr. BIEDERMANN'S *Technisch-chemisches Jahrbuch*, also for 1885. To purchasers of the *Chemiker-Kalender*, the *Jahrbuch* will be sold at a 20% discount. The price of the *Kalender* is three shillings. The ground covered by this work may be gathered from the following excerpt taken from the *INHALTS-VERZEICHNIS*:—Übersichts-Kalender für 1885. Häufig gebrauchte Atomgewichte und deren Multipla. Vorwort. Immerwährender Kalender, Zeit-tafel, Schreibkalender. Atomgewichte der Elemente, Volumgewicht, Volumgewichte von Lösungen: A, Alkalien; B, Säuren; C, Salze; D, Alkohole, etc., Löslichkeit, Eigenschaften chemischer Stoffe, Analyse, Maassanalyse, Spectralanalyse, Gasanalyse, Physio-logisch-chemische Analyse, Technisch-chemische Untersuchungen: I., Wasser; II., Probirkunst; III., Brennmateriellen; IV., Schwefelsäure; V., Alkali; VI., Chlorkalk; VII., Stärke und Zucker; VIII., Farben; IX., Keramik; X., Glas, XI., Mörtel und Cement, Mineralogie.

The Beilage zum *Chemiker-Kalender*, 1885, contains tables and data under the following headings: I., Münz-tabellen; II., Mathematik; III., Maas und Gewicht; IV., Wärme; V., Ausdehnung; VI., Dampfspannung; VII., Barometrie; VIII., Licht; IX., Electricität; X., Magnetismus; XI., Diffusion und Capillarität; XII., Volumgewicht. Anhang: (Für Studierende der Chemie wichtige Vorlesungen. Preise der wichtigsten chemischen Apparate) Anzeigen.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1884.

13923 W. Whitworth Nightingale, London. Improvements in the manufacture of transparent ice. October 21

13925 A. M. Clark, London. An improved process of, and apparatus for, purifying glycerine and other substances. Communicated by F. Howard Houghton, United States. Complete specification. October 21

13929 J. H. Johnson, London. Improvements in bleaching, and apparatus therefor. Communicated by E. Hermite, Paris. October 21

13940 W. R. Lake, London. An improved method of, and apparatus for, treating sugar cane preparatory to the extraction of sugar therefrom. Communicated by E. Schulze, Amsterdam. Complete specification. October 21

13961 H. Rees, Battersea. A method of preparing books to hold, convey, and exhibit chemicals, colours, and useful preparations, combined with instructions for their application. October 22

13978 E. W. Parnell, Liverpool. Improvements in the treatment of complex ores and substances containing antimony for the purpose of obtaining copper, lead, cobalt, and nickel, or one or more of these metals. October 22

14010 H. J. Cooper, London. Improvements in the manufacture of Portland cement. October 22

14011 J. Inray, London. Process for extracting and saccharifying ingredients of amylaceous substances by treatment with malt. Communicated by L. Cuisinier, Paris. Complete specification. October 22

11018 W. R. Lake, London. Improvements in apparatus for carburetted or enriching air, or impregnating the same with inflammable vapour, for illuminating or heating purposes. Communicated by P. T. Dubos, Rouen. October 22

11020 W. G. White, London. Improvements in colour printing, and in the preparation of colours to be employed therein. October 22

11022 T. Holliday, London. Improvements in dyeing vegetable or other textile fibre. October 22

11035 G. Quarrie, Liverpool. Improvements in the process of obtaining, and in the application of, a useful product or products from peat. October 23

11036 F. C. Cramer, Liverpool. Improvements in the manufacture of glue, applicable for use with tinfoil. October 23

11019 J. G. Willans, Westhamstead. Improvements in preparing iron ores and oxides in coking ovens for use in manufactures. October 23

11071 J. Makin, Manchester. An improvement in coating iron, steel, or other metal with lead. October 24

11083 W. Edwards, London. A new or improved material adapted and applied to the manufacture of life buoys, swimming belts, anchor buoys, and the like. October 24

11085 T. Hampton, London. Improvements in the manufacture of steel and ingot iron. October 24

11087 J. Farthing and J. H. Lorrimer, London. Improved manufacture of artificial asphalt. October 24

11091 H. W. Fenner, London. Improvements in stills or boilers for distilling tar or other hydrocarbon substances. October 24

11095 B. Peacock, London. Improvements in retorts for melting materials in the manufacture of glass. October 24

11111 F. Noel Mackay and W. Baxter, Liverpool. Improvements in apparatus for solidifying paraffin in oils. October 25

11110 B. Bröns, London. Improvements in the manufacture of explosive compounds. October 25

11148 F. Herbert, London. Improvements in the treatment of sewage, the said improvements being in part applicable to the purification of foul liquids. October 25

11152 A. G. Brookes, London. Improvements in the process of, and means or apparatus for, concentrating ore. Communicated by H. Pratt Tobey and G. Burder Thayer, United States. October 25

11158 W. R. Lake, London. Improvements in the manufacture of steel. Communicated by W. Courtenay, United States. Complete specification. October 25

11160 G. F. Redfern, London. Improvements in dyeing cotton. Communicated by E. E. Haffey, St. Die. October 25

11173 J. Waller Pratt, Liverpool. Improvements in purifying and decolourizing crude nitric acid, and in apparatus therefor. October 27

11175 R. Vaughan Dickens, Windsor. The application of glass or vitreous material to the construction of monumental tablets or records. October 27

11187 H. J. Allison, London. An improved medical compound. Communicated by E. A. Guptill, United States. October 27

11198 H. G. Fairburn, Cardiff. Improvements in machinery for washing, separating, and purifying coal and other substances. October 27

11199 H. G. Fairburn, Cardiff. Improvements in machinery for desiccating at low temperatures coal and other substances. October 27

11208 C. Gelstharp, London. Improvements in treating ores or compounds containing copper, iron, or sulphur. October 27

11225 J. Clare, Saddleworth, Yorkshire. A new process of dyeing black on cotton, cotton yarns, or cotton fabric, or other vegetable fibre. October 28

11210 A. M. Clark, London. Improvements in the preparation of caseine and in the product so obtained, and in articles made therefrom. Communicated by E. E. Childs, United States. October 28

11241 A. M. Clark, London. An improved composition mastic. Communicated by A. Denon, United States. Complete specification. October 28

11249 J. Nicholas and H. Lafone, London. An improved process for refining sugar. October 28

11252 C. M. Pielsticker, London. Improvements in the production of steel (and other metal) wire, plates, rods, bars, etc., and in apparatus connected therewith. Partly communicated by Dr. F. C. G. Müller, Prussia. October 28

11253 W. E. Gedge, London. Improvements in the manufacture of tin-plate. Communicated by The Société Générale des Cirages Français, Paris. October 28

11260 A. W. L. Reddie, London. Improvements in the manufacture of gas. Communicated by R. A. Chesbrough, United States. Complete specification. October 28

11261 S. Pitt, London. Improvements in the manufacture of iron and steel by the employment of a purified metal. Communicated by A. Rollet, St. Etienne. October 28

11276 W. P. Thompson, Liverpool. Improvements in dyeing with aniline-black. Communicated by A. Deseroix, Villefranche. October 29

11285 E. Brooke, Halifax. An improved construction of tank for melting glass. October 29

11289 J. Sundstrom, Bromley-by-Bow. Improvements in pans for evaporating cane juice, and like solutions or liquids likely to boil over. October 29

11291 T. F. Haldane and W. D. A. Bost, Glasgow. Improvements in the destructive distillation of shale, and in the apparatus employed therefor. October 29

11308 C. Leech, T. Neal, O. M. Lilly, and H. J. Staples, London. Improvements in the manufacture of Indian red, Venetian red, and other oxide of iron pigments. October 29

11322 A. M. Clark, London. An improved process of, and

apparatus for, decolourizing, clarifying, and rendering limpid on solution in the cold, tanning extracts and liquors used in tanning and dyeing. Communicated by J. Serriere, Lyons. October 29

11321 P. M. Justice, London. Improvements in furnaces for burning cement and other substances, and in the linings therefor. Communicated by C. Dietrich, Malsatt. October 29

11326 I. Levinstein, London. Improvements in the manufacture of colouring matters. Communicated by Dr. H. Reinberz, Hanover. October 30

11328 C. Thompson, Sunderland. An improved form in bricks for the construction of furnaces, stoves, kilns, etc. October 30

11358 E. Capitaine, London. Process for the manufacture of celluloid or similar materials. Communicated by Rheinische Hartgummi-Waaren-Fabrik, Mannheim. October 30

11361 M. Cock, London. An improved coating for explosive compounds and cartridges. Complete specification. October 30

11365 W. Dawson, London. Improvements in mills or triturators for reducing cement or other hard substances. October 30

11379 F. M. Lyte, Putney, Surrey, and C. L. J. A. Lewall, London. Improvements in the manufacture of explosive compounds. Oct. 30

11393 J. Stansfield, Fleetwood. Improvements in the manufacture and laying of asphalt. October 31

11427 F. Arnott, London. Improvements in apparatus to be applied in connection with coke ovens for conducting away from such ovens the gases, and for receiving the condensable constituents as they separate therefrom. October 31

11430 A. Bruce Joy and J. J. Lewen, London. An improved process for making imitation marble. October 31

11466 W. Briggs, London. Improvements in hand grenades for extinguishing fires. November 1

11468 C. Thompson and C. Hammond, London. Manufacture of "Virgin Gas" for lighting, heating, and other purposes. November 1

11509 L. A. Groth, London. Improvements in gas-producer furnaces. Communicated by W. Backer, Budweis. Complete specification. November 3

11530 W. P. Thompson, Liverpool. Improvements in the arrangement and construction of refrigerating chambers or buildings. Communicated by J. F. Hanrahan, United States. Complete specification. November 4

11533 H. Whitehead and R. Hodgson, Liverpool. Improvements in the manufacture of salt from brine, and in apparatus therefor. November 4

11543 G. Washington Mears, London. Improvements in smoke consumers. Complete specification. November 4

11549 P. M. Justice, London. Improvements in the treatment of alkaline chlorides. Communicated by L. E. Thomas, Paris. November 4

11551 J. Jameson, London. Improvements in and apparatus for the manufacture of coke. November 4

11553 G. Davidson, R. Cattley Jackson, and J. Black Duncan, London. Improvements in the manufacture of carbons for incandescent electric lamps and apparatus to be used therein. November 4

11556 H. Simon, London. Improvements in covers for retorts and similar receptacles. Communicated by the Berlin-Anhaltische Maschinenbau-Actien Gesellschaft, Berlin. November 4

11561 J. Doutreleau, London. Process for discolouring and purifying vegetable extracts containing tannin. November 4

11563 W. White, London. Improvements in the manufacture or production of aluminium chloride. November 4

11561 E. de Pass, London. A process of manufacturing tubes in steel, ordinary iron, homogeneous iron, alloyed iron, pure or alloyed copper, and all other pure or alloyed metals. Communicated by P. E. Secrétan, Paris. November 4

11587 A. M. Clark, London. An improved manufacture of artificial leather. Communicated by H. Schwabacher, Paris. November 4

11596 W. Tait and A. Lilley, Burton-on-Trent. An improvement in coolers, refrigerators, and condensers. November 5

11603 J. Plummer, Glasgow. Improvements in obtaining sulphur from alkali waste. November 5

11607 S. Turner and J. Bull, Manchester. Improvements in the manufacture of asbestos-metallic cloth. November 5

11609 J. Robertson, Manchester. Improvements in apparatus used in the manufacture of paper. November 5

11621 J. Hatfield, London. Improvements in the manufacture of artificial stone. Complete specification. November 5

11633 C. Cochrane, London. Desiccation of the air supply of furnaces, and apparatus for drying air for that or other purposes. November 5

11672 H. J. Haddon, Westminster. Process for the recovery of tin from tin-plate scrap, and other materials containing tin, such as oxidised metal and residues. Communicated by A. Lambotte, Brussels. November 6

11673 E. H. Remmers and J. Williamson, London. An improved process of filtering paraffin oils, and of refining the solid paraffin obtained therefrom. November 6

11681 C. R. Alder Wright, London. Improvements in the manufacture of soap. November 6

11680 F. J. Brougham, London. Improvements in the process of and means for equalizing the temperature in cast-steel ingots. Communicated by A. Sailer, Witkowitz. November 6

11694 T. Horsburgh, Birmingham. A new or improved machine for the preparation of finings for beer and other liquids. November 7

11700 W. P. Thompson, Liverpool. Improvements in the distillation of fatty substances and the like by means of super-



heated steam and apparatus therefor. Communicated by M. Julien and M. Blumski, Odessa. November 7

11712 J. N. Mocrath, London. Sanitary constructions of fire and earthquake-proof houses or other buildings. November 7

11721 H. E. Devaux, London. Improvements in the manufacture of cement. Communicated by J. Farinaux, Lille. November 7

11725 A. J. Boulton, London. Improved process and solution or liquid for treating textile and other materials whereby they may be rendered waterproof. Communicated by O. B. E. Müller, Berlin. November 7

11734 J. Anderson, Broughty Ferry. Improvements in the manufacture of sodium and potassium. November 8

11735 T. Graham Young and J. Pettigrew, Glasgow. Improvements in treating vegetable substances in order to obtain fibrous materials for textile purposes, and pulp for making paper, and for other useful applications. November 8

11736 J. Riley and W. Crossley, Glasgow. Improvements in the manufacture of steel, and in furnaces for melting and treating iron, steel, or other substances. November 8

11737 E. J. Mills, Glasgow. Improvements in the formation or preparation of explosive charges. November 8

11753 C. Havens Royce, London. An improved fire-extinguishing compound. November 8

11770 G. E. Ellis, London. A new or improved manufacture of filtering, disinfecting, deodorizing, and decolorizing medium. Complete specification. November 8

11771 A. H. Reed, London. A new or improved apparatus for the manufacture of a lighting gas from hydrocarbons. Communicated by A. Michaux, Paris. November 8

11776 A. Parkes, London. Improvements in the production of compounds capable of being employed for coating metallic and other surfaces. November 8

11785 H. Howlett Redfern, Hanley, Staffordshire. Improvements in kilns for calcining flints, felspar, stone, and other materials, with either coal, coke, or gas. November 10

11790 J. Rushton Turnock, Loughor, South Wales. A movable tyre or rable for the purpose of conveying air, gas, or steam below the surface of molten metal. November 10

11792 D. Dawson and H. Broadbent, Halifax. New or improved apparatus for heating dye pots by means of steam, such apparatus being specially applicable for experimental dyeing in industrial schools and laboratories. November 10

11810 J. H. Weston, London. Improved arrangement of apparatus connected with increasing the brilliancy of coal-gas by the admixture of vapour from a liquid carbon. Nov. 10

11822 J. Yate Johnson, London. Improved means for the purification of water. Communicated by A. R. Leeds, United States. November 10

11833 A. W. L. Reddie, London. Improvements in treating copper matt. Communicated by J. J. Crooke and R. Crooke, United States. Complete specification. November 11

11859 H. Vessier and A. Wilbaux, London. An improved cold process for the manufacture of paper pulps and bleached or unbleached textile fibres, and apparatus therefor. Nov. 11

11860 C. Cheswright, London. Improvements in the manufacture of metal foil, and in the apparatus or means employed therein. November 11

11861 J. Y. Johnson, London. A new or improved disinfectant. Communicated by E. Otho Beck, Paris. Nov. 11

11866 F. Bolton, Knight, London. Improvements in the manufacture of caustic soda and potash, and by-products therefrom. November 11

11908 George Harrison, London. Improvements in the proofing of textile fabrics. November 12

11911 Lieut.-Col. W. Hope, London. Improvements in gunpowder for ballistic purposes. November 12

11915 Lieut.-Col. W. Hope, London. Improvements in ammunition for military and sporting small arms, and field and machine and other guns. November 12

11930 H. Stone, London. Improvements in smelting, and in apparatus connected therewith. November 12

11967 C. E. Layton, London. Improvements in the manufacture or production of a substitute for lithographic stones. Partly communicated by P. Corlitz Moller, Leipsic. Nov. 13

11988 T. Graham Young and J. Pettigrew, Glasgow. Improvements in treating vegetable substances in order to obtain pulp for making paper, and for other useful applications. November 13

11989 R. Leigh Lowe, Manchester. Improvements in cement or composition for use in the laying of floors and pavement. November 13

11992 J. A. Elliott, London. Improvements in apparatus for sulphurizing and phosphorizing friction matches. Communicated by E. Fitch, Canada. Complete specification. Nov. 13

15010 J. C. Mewburn, London. An improved manufacture of non-conducting material for covering steam boilers, steam pipes, hot or cold water pipes, and for other like purposes. Communicated by J. L. Martiny et Compagnie, Paris. November 14

15013 J. P. Malvin Millard, London. A process for producing a coating for protecting metal goods against rust or oxidation. Communicated by C. Haselhorst, Dresden. November 14

15011 J. Thompson and J. H. Bryant, London. An improved artificial stone. November 14

15018 A. C. Henderson, London. Improvements in the manufacture of baryta and strontia. Communicated by J. Martin and E. Maumene, Lyons. November 14

15026 L. Quentin Brin and A. Brin, London. Improvements in the separation and obtaining of oxygen and nitrogen from atmospheric air. November 14

15027 F. J. King, London. Improvements in the separation of ores and mineral products, and in apparatus employed therein. November 14

15029 J. Sheldon, London. Improvements in the manufacture of polished and annealed wire or sheets of iron or steel, partly applicable to annealing other articles of iron or steel. November 14

15031 C. Rumble and F. Sear, London. Improvements in the preparation of oils used for lubricating wools preparatory to spinning. November 14

15034 W. Macrone, Glasgow. The application of waterproofing to paper and other fabrics. November 15

15036 B. W. Goodison, Liverpool. Improvements in the manipulation of cements to form a composition which shall be "stonecrete." November 15

15038 F. Seltam, Liverpool. Improvements in the process of extracting and drying bones and the like. November 15

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## OF THE

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### NOTICES.

The Council of the Society, at their last meeting, had under consideration the advertisement of Mr. G. E. Davis, late Alkali Inspector, which appears in the Journal of the Society. The Council passed unanimously a resolution disapproving of the terms of the said advertisement, but regrets that it has no power to order its rejection owing to a contract with the printers, which is binding until the 31st December, 1884.

In reply to numerous inquiries the Secretary begs to inform Members that the first six numbers of the Society Journal for 1882 have now been reprinted, and can be obtained on application.

The Proceedings of the First General Meeting (1881) of the Society of Chemical Industry have been reprinted in such size and style as to permit of their being bound up with the Journal. Copies of the reprinted Proceedings will be forwarded by the Publishers on receipt by them of twelve penny stamps for each copy required.

**NOTICE TO MANUFACTURERS AND OTHERS.**—In consequence of numerous inquiries, attention is called to the fact that the price of extra sets of the Journal to members is one guinea, whether such sets are for the current year or for past years. A misapprehension on this score appears to have deterred certain members from applying for duplicate copies for official and laboratory use.

Authors of papers printed in the Journal are hereby notified of the fact that, in accordance with Bye-law 43, they are entitled to receive not more than 50 gratuitous copies of their papers. Authors should state on their manuscripts their desire to have free copies, adding the number wished for. Unless the contrary be specially desired, this being stated on the manuscript, the reprints of an author's paper will not include the report of any discussion that may have arisen after the reading of the paper.

In view of the approaching issue of a revised list of Members, the Secretary trusts that Members will assist to make this as complete as possible, and obviate the necessity of a supplement, by furnishing at their earliest convenience their existing and prospective changes of address.

Those Members who have not yet paid their Subscription for 1884 are requested to send to the Treasurer a remittance at once.

Members are hereby advised that the Subscription for 1885 falls due at the beginning of the year, and should be paid as soon afterwards as possible. The Council has decided that after January 1st, 1885, the 29th Bye-law will be strictly enforced, so that Members whose subscriptions are more than four months in arrear, will not continue to receive the Society's Journal.



Members are requested to take notice that Mr. Ludwig Mond, Honorary Foreign Secretary of the Society, has removed from Winnington Hall, Northwich, to 28, Park Crescent, Portland Place, W.

The Council begs to call the attention of Members of the Society to the following Circular, which has been issued by a committee formed for the purpose of raising a fund for the benefit of the widow and family of the late Mr. HENRY WATTS, F.R.S.—

#### PROVISIONAL COMMITTEE.

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It having become known to some of the friends of the late Mr. HENRY WATTS, whose death occurred very suddenly on the 30th of last June, that his wife and family are in very straitened circumstances, an informal meeting was held at the Royal Institution on Tuesday, the 11th of November, 1881. Those present resolved to form themselves into a Committee, with power to add to their number, in order to collect a fund for the benefit of Mrs. Watts and those of her children who are not of an age to provide for their own support. Dr. Atkinson consented to act as Secretary, and Dr. Perkin, President of the Chemical Society, as Treasurer.

Mr. WATTS's public labours for the advancement of Chemical Science may be said to have begun with the translation of Gmelin's "Handbook of Chemistry," the admirable English edition of which was prepared and edited for the Cavendish Society by him. This work occupies eighteen large octavo volumes, of which the first appeared in 1819, and the last in 1871. A work scarcely, if at all, inferior to this in magnitude, and one which has perhaps been of even greater service to English chemists, both scientific and industrial, is Watts's great "Dictionary of Chemistry," which appeared from 1863 to 1881, in eight volumes, containing altogether nearly 9,700 pages. Mr. WATTS also edited and largely added to the second volume of the late Professor Graham's "Elements of Chemistry," published in 1858; he prepared several editions of Powne's well-known "Manual of Chemistry," which he almost entirely rewrote and made into virtually a new work; and in conjunction with Mr. Ronalds and Dr. Richardson he prepared for Messrs. Baillière an elaborate treatise on Chemical Technology. Up to the time of his death, and for about thirty years previously, Mr. WATTS was Editor of the Journal of the Chemical Society, and in this capacity, as well as in that of Librarian to the Chemical Society, he became personally known to and gained the friendship of very many among the Fellows of the Society.

But although Mr. WATTS's life was one of unremitting labour, the money return for his work was barely sufficient to enable him to provide for the daily wants of a delicate wife and a numerous family. It was not possible for him to provide for their future needs. But if he could not leave behind him pecuniary resources, he accumulated esteem and affection among all who knew him, which, it is confidently hoped, will prove a valuable legacy for those who were dependent on him. The following facts will show that there is great need of whatever practical proof of their regard for him and appreciation of his labours Mr. WATTS's friends, and English chemists generally, may be willing to make.

For many years Mrs. Watts has been in ill-health, so that she cannot do anything for her own support and that of her family. Her only income is about £100 a year, and of her ten children only two are in a position to afford her help. They hope to contribute between them £150 a year. One son is a permanent invalid, and the four youngest children have still to be educated. A considerable expenditure is therefore unavoidable for a good many years to come, if the children are to have a fair chance of a start in life.

Subscriptions will be received and acknowledged by the Secretary.

Dr. EDMUND ATKINSON, *Portesbury Hill, Camberley, Surrey,* or by the Treasurer,

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MEETINGS, SESSION 1884-5.

### PROVISIONAL PROGRAMME.

January 5.—C. N. Cresswell, Esq. (barrister-at-law), on "The proposed Rivers' Pollution Bill." Messrs. Cross and Bevan on "Cellulose and Hydration." Professor Munroe: "Composition and Manurial Value of Filter Pressed Sewage Sludge."

February 2.—Mr. Boverton Redwood on "The Russian Petroleum Industry."

March 2.—Mr. W. J. Kemp: "Some Experiments upon that part of the Patent of Mr. F. B. Rawes for the 'Recovery of Sulphur,' etc., etc., which depends upon the Action of Carbon Dioxide upon Soda Waste Suspended in a Liquid." Mr. W. J. Williams on "The Treatment of Certain Phosphatic Minerals."

April 13.—Mr. W. J. Dibdin—"Further Notes on the 'Radial' Photometer, and the Proposed Standards of Light."

May 4.—Mr. G. H. Beckett—"Hempel's Method of Gas Analysis." Messrs. Giles and Shearer—"The Real Strength of Aqueous Solutions of Sulphurous Acid."

June 1st.—

July—Annual General Meeting of the Society.

The paper on "Photography," read by Mr. J. M. Thomson, on December 1st., will be printed in a subsequent number.

## ERRATA.

In Mr. Deering's paper on some points in the examination of tallow and some commercial oils, especially for acidity, November number.

Page 541, Tallow, No. 23.—For 39°47' read 0°47'  
" 543, Line 39.—Omit "unboiled."

## Liverpool Section.

Chairman: E. K. Muspratt.

Vice-Chairman: J. Campbell Brown.

### Committee:

Ernest V. Bibby.	C. Symes.
Hudson A. Binney.	G. I. J. Wells.
Enstace Carey.	F. Hurter.
John Hargreaves.	H. Brunner.
E. Milner.	A. Norman Tate.
E. W. Parnell.	J. Affleck.

Local Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be sent to the Local Secretary.

The next meeting will be held on January 7th, 1885, when Dr. Hurter will read a paper on "The Advisability of Adopting the Metric System of Weights and Measures in this Country."

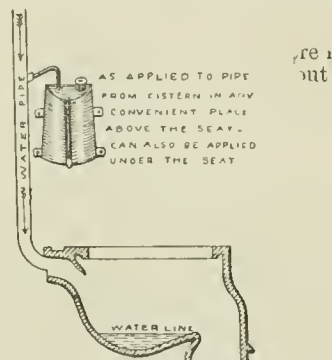
UNIVERSITY COLLEGE, ASHTON STREET, LIVERPOOL,

Wednesday Evening, Nov. 5th, 1884.

## A NEW APPARATUS FOR DISINFECTING PURPOSES.

BY T. WILLIAMS, F.C.S.

THE apparatus which I have the pleasure of exhibiting to you this evening, is a patented appliance for supplying automatically, disinfectants to water-closets, slop-stones, baths, etc. The principle upon which it works is very simple being a vacuum or water pump, the head of water obtained from the waste water preventer cistern, passing down the flush pipe connected with the water-closet, creating a suction in the small tube attached to the disinfecting vessel, carrying over each time sufficient disinfectant to prevent decomposition. The moment that the water ceases to flow the small tube inserted in the disinfectant vessel becomes a syphon and draws back the liquid disinfectant remaining in the tube. The glass tube in the centre of the vessel shows the amount of liquid disinfectant in the apparatus, so that no difficulty can arise in



knowing the quantity. The apparatus having no valve, tap, lever or other working parts, cannot possibly get out of order, the action being governed entirely by a natural law. The apparatus is adapted for the use of any liquid disinfectant, such as carbolic acid, Condy's fluid, chloride of zinc; but is now charged with a new disinfectant called "THYMO CRESOL," a preparation from tar acids which obtained the gold medal at the London International Exhibition in August last, and has been certified by many high authorities to possess extraordinary germicidal properties and is a very active antiseptic agent. The great miscibility of this preparation with water and being non-poisonous and non-corrosive entitles it to the foremost rank amongst disinfectants.

The very easy manner in which this apparatus can be applied to water carts, flushing tanks etc., is to my mind a great recommendation and that the utility of such an appliance cannot be doubted the disinfectant being automatically and systematically delivered and when universally adopted would entirely prevent the formation of sewer gas and the creation of myriads of disease germs. The sewage matter passing as it now does into our rivers, floating backwards and forwards with the rise and fall of the tide would not throw off the vile exhalations which we now feel almost always in the Thames, and frequently here in the Mersey. The apparatus is specially adapted for large passenger steamers having no working parts that can get out of order, it will work in any position of the vessel, and the motion of the vessel cannot



cause any derangement of the automatic action. For cattle ships the apparatus being fixed to the hose-pipe, all parts of the vessel can be equally sprinkled with any required quantity of disinfectant and in a much surer and more efficacious a manner than by the use of any powder disinfectants.

## Manchester Section.

Chairman: Sir H. E. Roscoe.

Vice-Chairman: D. B. Hewitt.

### Committee:

R. F. Carpenter.  
C. Estcourt.  
H. Grimshaw.  
Peter Hart.  
J. von Hohenhausen.  
T. Jackson.

J. Knowles.  
I. Levinstein.  
C. Schorlemmer.  
Watson Smith.  
Wm. Thomson.  
D. Watson.

### Local Secretary:

J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

Notices of papers and communications for the meetings to be sent to the Local Secretary.

*Tuesday, November 25th, 1884.*

MR. H. E. ROSCOE IN THE CHAIR.

## REMARKS ON THE TEACHING OF CHEMICAL TECHNOLOGY.

### CHAIRMAN'S ADDRESS.

As the subject of the methods of teaching chemical technology has recently attracted attention in this Society, I do not know that I need apologise to the section for bringing before them my own views on the question.

In the first place we shall do well to get very clear ideas as to what we mean by this term, and what we propose to ourselves to accomplish, and in the second place let us understand whether this is practicable, and if so how we are to set about it.

It is perhaps more easy to give a negative than a positive definition of the term. No one who knows anything about the subject would support the proposition that the object of teaching technology is to qualify a man completely to manage an alkali works, or an alizarin works, or a print works, or a blast furnace when he leaves the technical school. Such an idea is absurd on the face of it, and needs, I take it, no further demonstration to a society like ours. All that such instruction can with any degree of success attempt is to lay a scientific foundation upon which the pupil is afterwards to build.

But that foundation may be more or less complete; if our building is to be stately and permanent, the foundations must be deeper and more extensive than when a less important edifice is to be reared.

In our case the first thing to ensure is that the student has obtained a careful and systematic grounding in the principles of the science the application of which it will be his business to carry out.

As Frederick the Great said that the three great requisites in war were money—money—money, so we may say that for technologists the three great requirements are science—science—science. But science is only knowledge systematised, and instruction only ceases to be scientific when it ceases to be systematic. Therefore, whilst I uphold that the only true way of teaching is reference to principle, I by no means am of

opinion that our teaching should be confined to theory, or to theoretical explanations of phenomena or processes which have no technical bearing whatsoever. On the contrary, I hold that after a student has mastered the A B C of our science, when he has, let us say, attended a course of experimental lectures on elementary chemistry, and has worked at ordinary qualitative and quantitative analysis, so as to give him an insight, first into the great principles of the science, and then into the chief properties and reactions of the more important elements and their compounds, and having obtained a notion of manipulation and a knowledge of what accuracy in scientific work means, I think it very desirable that, supposing him to be intended for a chemical manufacturer, he should attend a course of lectures on some specific subject connected with industrial chemistry. For instance, let him hear in detail the principles involved in the manufacture of sulphuric acid; let him have the chief points in the construction of the plant brought before him. Make him familiar with the causes of failure of the process, with the limits of error attaching to the reactions occurring on a large scale. Inform him of the weak points of the methods employed, and how these are to be remedied; let him understand how the various stages of the reaction can be watched and followed; explain to him by what means waste can be detected, and what the maximum practical yield ought to be, as distinct from the theoretical. Make him understand how waste products have gradually become valuable material. Give him, in fact, such instruction as will enable him intelligently to follow the manufacture when he is called upon to study it in the works. Above all, train him in habits of precise scientific thought, thus giving him some power, not only of detecting faults or accidents when they arise and of successfully combating them, but even of initiating improvements or new processes by a complete understanding of the old ones. The student of average intelligence having attended such a course—some 12 or 15 lectures, such as I have referred to on the special chemistry of sulphuric acid, should be able to answer such questions as:—

“Describe the various conditions of inlet and outlet for gases and air necessary for obtaining the maximum of work from a series of chambers.”

Or “Calculate the amount of air in cubic feet which must be supplied per ton of ore so as to have the necessary excess of about 10% of oxygen in the outlet.” Instruction giving information of this kind is strictly scientific, as long as it is systematic, and yet it is certainly of practical utility to any one who may in after years have charge of a works.

At this point I may refer, as a model of the scientific treatment of a technical subject, to the admirable lecture on the Manufacture of Sulphuric Acid by Dr. Hurter, delivered in Widnes in 1882.

From the study of the sulphuric acid manufacture the student may pass to that of the kindred subject of alkali manufacture, which is brought before him in a similarly connected and systematic manner.

And here I would have it to be understood that concurrently with lectures of the kind I have described, extended laboratory work is essential. Having learnt what exact quantitative work means, and having had experience in the separation of the more important metals and acids and their gravimetric determination, the student passes to volumetric analysis, and learns what degree of accuracy can be reached by these processes.

After having attended such a course of lectures as I have described, and after having gone through a satisfactory course of quantitative analysis, the student is in a position to test the value of the methods

of quick commercial testing used in the works, to understand to what extent they are reliable, and to check them by the more perfect but more lengthy processes with which he has been made familiar. Moreover, he is now able to appreciate and examine for himself some of the more abstruse reactions occurring in the manufacture: he can follow the complicated reactions taking place in the black-ash vat; he sees, for instance, the meaning of Pechiney and Weldon's process for the oxidation of the cyanides to cyanates, and the decomposition of these, with evolution of ammonia, and the value of the subsequent addition of limestone to convert the sulphide into carbonate. He should also be informed as to the use of the various gasometric methods employed in determining the acidity, or the amount of oxygen or carbonic acid contained in the exit flue gases, and learn something about the requirements of the Alkali Acts.

Who can doubt that an intelligent student, having received a training of which the above is a mere sketch, will be of more use in an alkali works than one who has not supplemented his purely scientific knowledge by some amount of detailed information as to the applications of that knowledge to his special branch of manufacture?

But I have hitherto only mentioned one branch of chemical industry. There remain many others, as, for instance, all the important applications of organic chemistry to the arts and manufactures. Can these be similarly dealt with in a technical course so that, still preserving a scientific treatment of the subject and building on his theoretical knowledge, the student may gain experience which shall be of value to him in after life? There is no doubt that this is both feasible and of distinct advantage to the pupil.

The subject of applied organic chemistry may, as is done in this college, be conveniently divided into three sections, each to be treated in a separate course of lectures: (1) The Chemical Technology of fuel including gas-manufacture, recovery of volatile products from this process and from coke-ovens and blast-furnaces, including the distillation and separation of the crude tar products; (2) The chemical technology of pure coal-tar products, especially the colouring matters; (3) The chemical technology of the application of colour to textile fabrics, including dyeing, bleaching and calico printing.

But before he is able to appreciate the first course, and to a still greater degree the second and third, the student must gain a mastery over the principles and chief facts of theoretical organic chemistry. He cannot understand the methods used in the separation of the numerous constituents of coal-tar unless he knows the distinctive properties of the paraffins, as against the olefines or the benzene series of hydrocarbons.

Here again—science—science—science—is the one necessary preliminary, and here it is that we English are apt to make the great mistake of underrating the value of and ignoring the necessity for theoretical knowledge, and it is in the gradual appreciation by all classes, from masters to men, of this necessity that we can hope for an improved state of things.

In this branch again, an extended course of laboratory work must be carried on concurrently with the lectures on pure organic chemistry, and one or other of the above technological courses. The student must learn what accurate work, this time in the organic branch, means; he must obtain facility in making exact organic combustion analyses, in determinations of vapour density, in fractional distillation, and especially in the preparation of pure organic compounds. He may then pass to some easy investigation of a kind which gives him an insight into some of the processes now conducted on a large scale either in tar-distilling or colour-making. Thus he may prepare

the three isomeric xylenes from crude naphtha, separating the meta-compound as being the one used in commerce and estimating its quantity.

Or he may make a careful examination of the various constituents of coke-oven-tar, comparing these products with those derived from ordinary coal-tar—a most useful and instructive problem.

Again, he may prepare some of the azo-naphthol colours, beginning from the hydrocarbon naphthalene, and working quantitatively up to the colour, or again, he may work up to eosine from benzene and naphthalene, or prepare alizarin from anthracene. Scores of other examples may be found equally well suited for the instruction and practical experience of the student.

Next, as regards the technology of bleaching, dyeing, and calico-printing I wish again to point out still more strongly the necessity of a sound basis of theory. For here we have arrived at the most complicated series of operations. Not only must we know the properties of our bleaching agents and dyes and colours, but we must be acquainted with their action on textile fabrics and the proper mode of their application and their mutual reactions.

Here, too, mechanical appliances and the necessity of meeting practical exigencies come into play more than in perhaps any other branch of applied chemistry. So that the difficulties of the teacher of technology are much greater, and much more must of necessity be left to the student to work out in his subsequent experience in the works. But even admitting all this, much can be done to place the principles and chief facts before the pupil in the technical school.

As regards the mode of teaching the principles of dyeing, I think that a scheme of scientific instruction is quite possible, that, for example, a scientific method of examining the effects of various mordants on certain textile fibres in conjunction with those of certain dyes, according to a graduated and quantitative method, would be scientific in principle and instructive and valuable in result. But such a course of instruction, which is more closely allied with every day work and practice in the dye-house and dye-house laboratory (or ought to be with regard to the latter), should certainly for our chemical students follow, and so be subservient to, the more theoretical courses of scientific instruction, in which principles are dealt with. The foundation of scientific principle and fact must first be laid, and time must be allowed for this, before methods of dealing with and examining practical or technical details of processes carried out in the factories, however scientific they may be, are gone into. "There is a time for everything," and with regard to the time devoted to study by students of chemistry, which in this country is truly short, and exceedingly so compared with that of the whole after-career devoted to the acquiring of the practical details, *it is a question* how far that after-career should be as it were, anticipated in the already too-short time allowed for the grounding in principles. I think *it is possible* to encroach too much on time allowed for the study of first principles, to the detriment of that study, and the consequent diminution of future possibilities of application of theory to practice. Of course granted sufficient time, every branch may then be adequately acquired and learnt.

I do see a danger which must be guarded against of making the instruction merely a routine and rule-of-thumb one. If the student possesses a good theoretical and practical knowledge of his science, the systematic preparation in the dyeing laboratory of a series of dyed samples of wool, silk, or cotton, according to a variety of receipts, may be of great advantage, as giving him a general view of the



methods of dyeing as carried out on the large scale. But unless he knows the nature, composition, and properties of the various colouring matters he uses, and has had a thorough grounding in chemical science this must degenerate into working to pattern, and cannot be said to encourage an insight into the processes, to incite inquiry as to causes of failure, or to give a training which shall in any degree fit the pupil to overcome difficulties, much less to enable him to strike out in a new direction.

A course of lectures on these subjects to well-grounded students, properly illustrated by specimens, experiments, and models, cannot, I think, fail to be of service. And we in this Society may well bear in mind that our Journal, bringing, as it does, every month accounts of all the important recent improvements and novelties in every branch of chemical industry, serves as a kind of text-book of chemical technology, useful alike to teacher and learner. To quote only one instance, I may remind you first of the abstract in the October number of 1883, and afterwards of the paper read before this Section by my friends and former pupils, Messrs. Cross and Bevan, in which the interesting process of bleaching by carbonic acid in conjunction with chloride of lime was fully described, and the important bearing on this subject of Witz's researches on oxycellulose, and those of the authors themselves on the constituents of bast fibre was clearly shown. In a similar way lectures, accompanied by practical laboratory work, on the chemical principles involved in brewing and in many other separate branches of applied chemistry may with advantage be given to those who desire to qualify themselves for such branches. But to erect brewing-plant or a salt-cake furnace in a school, or to endeavour to work metallurgical processes on a large scale, or even to dye or print in quantity, would, I believe, be a step neither of commercial nor of educational advantage.

I am perfectly aware of the difficulty of obtaining properly qualified teachers in these several branches, and I know well that even competent men who have had both a theoretical, or purely scientific training, and practical experience in works, find it extremely difficult to make themselves acquainted with the newest steps in the varying and progressive processes of applied chemistry. This knowledge can only be gained by practical experience and by intimate contact with industrial life. But though these circumstances may render it impossible to teach chemical technology completely up to the standard of the most advanced manufacturers of the day, there is surely still plenty of room left for the teacher.

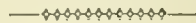
We may indeed be well content if our students are brought so near the harbour for which they steer, that they can recognise and appreciate the value of the new lights which by degrees open out to them in practice. We may be satisfied if by careful training they are brought to know the principles of their calling, though their knowledge of detail be deficient, and even somewhat antiquated.

Hitherto I have been speaking of what may be termed the rank and file of our young technologists—of those who, having perhaps only a moderate amount of ability and also only a moderate supply of money, and therefore of time to spend on their education, are anxious to make the most of their opportunities, and desire to enter upon remunerative employment in works as soon as possible.

I should like now to express my opinion that the course which I have sketched, though sufficient perhaps for the above class, is insufficient for those brighter and more capable—and, perhaps, more fortunate—persons, who look forward to becoming our leaders in industry, who are determined to push

beyond the routine of the present, to initiate new processes. Such persons must carry on their scientific education to a higher level; they must learn in the greatest of all schools, that of original research, for in that alone will they obtain that self-reliance, that independence of thought, and that power of overcoming difficulties, which are needed for carrying out any new and successful enterprise. The problems which present themselves for solution to the student who undertakes original scientific work in the laboratory are, so far as the chemical portion of the work is concerned, on a small scale analogous to those which he has to meet on the large scale in the works. If he has learnt the methods by which the one can be overcome, he is in possession of the means of vanquishing the other.

Of course I am aware that another important subject forms an essential part of the education of a chemical technologist, namely, that of engineering. Time will not permit me to discuss at length how much of this branch of applied science ought to be taught to the chemical student during his college career. But I think all will admit at once that a course of theoretical lectures, accompanied by instruction in mechanical drawing, must be of great value to the manufacturing chemist. So strongly indeed do I feel this that in our system of instruction in chemical technology in the Owens College, this latter subject is made obligatory. Then, again, the value of a general preliminary literary and scientific training in such subjects as mathematics, physics, and mechanics, and, though last not least, the modern Continental languages, is patent to all. But the great difficulty which meets us on every hand is the proneness of the English mind to look for immediate results. We are proud of being called a practical nation; let us take care that with this we unite theory or scientific knowledge, and all will be well with us; whilst if we continue to be content with practice alone, we shall find ourselves outstripped in our industries by those who see more clearly than we do in what the real strength of a manufacturing nation lies.



## ALIZARIN IN WOOL-DYEING.

BY J. J. HUMMEL, F.C.S.

*Dyeing School of the Yorkshire College, Leeds.*

COULD we devote sufficient time to consider at length the history of dyeing we should be struck with the very great assistance which it has derived from chemical science, especially in these latter days. As it is with most other arts, the origin of dyeing is shrouded in the obscurity of the past, and I am not quite sure whether the fact that this art had attained a most venerable age long before the science alluded to was even dreamt of, has not something to do with the silent indifference which the dyer sometimes manifests towards the teaching of the chemist whenever the latter deals with matters tinctorial.

Let me remind you that we owe the publication in 1662 of the first account in the English language, of the methods and means employed in dyeing, to a very august body of scientific men, none other indeed than the Royal Society of London. As you know full well, there was a time when the dyer derived his colours almost entirely from the vegetable kingdom, but in 1834 an indication of a coming different state of affairs appeared in the scientific researches of the celebrated German chemist Runge, and although the memorable discovery in 1856 of the first aniline dye (mauve) by the English chemist Perkin, was, as we learn from his own lips, quite unexpected,

and in so far accidental, we also know that theoretical considerations were nevertheless involved, and it is undoubtedly to the prosecution of scientific research that we owe the discovery that coal-tar is such a rich mine of wealth in dyeing materials as it has now proved to be. It would take too long to allude further to the discovery of these colours, but the result of the labours in this direction has been, as you know, that we are now able to obtain from coal-tar all the colours of the rainbow. On my right we have a list of the most important of these colouring matters, with the dates of their discovery and the names of their discoverers, a list representing to a certain degree the practical result of the scientific work in this department during the past 28 years. You see there too a list of the principal natural colouring matters previously known, accumulated during thousands of years, since the very beginning of the dyeing art in fact, and comparing the two lists, the dyer cannot but admit that science has been his friend. I am inclined to think that many people at present regard these so-called "aniline dyes" as colours which are certainly very brilliant, but which soon fade, or wash out. In the early days of the aniline colours this opinion was undoubtedly correct, but to-day it would be quite a mistake to make any sweeping declaration of this kind. Science is progressive, and of late years, chemists have entered new fields of discovery among the coal-tar colours. Not only do some of the recently discovered colours combine fastness with their brilliancy, but two of them are identical with time-honoured colouring matters obtained from plants. The most valuable and permanent of these, the very strongholds of nature, so to say, have been the first to succumb to the vigorous and oft-repeated attacks of the chemist. I refer, of course, to indigo and alizarin, and it is to the latter of these that I would wish to direct your attention for a short time this evening.

What is alizarin?

Alizarin is the pure colouring principle which exists to a very small extent in the madder-root, and to which the latter owes its dyeing properties.

After many years of patient waiting and laborious yet loving research, it fell to the lot of two young German chemists, Graebe and Liebermann, to discover how it might be prepared artificially from the coal-tar product anthracene.

It is not my intention to say anything about the method of manufacturing alizarin, I would simply refer you for information on this point to the excellent and interesting paper on the alizarin industry, read in May last by Mr. I. Levinstein before this Section.

It is necessary, however, that I should remind you that Graebe and Liebermann's discovery included, or rather led to, the recognition of three distinct though closely allied colouring matters, viz., *alizarin*, *isopurpurin* or *anthrapurpurin*, and *flavopurpurin*.

Another closely allied colouring matter which exists in the madder-root, viz., *purpurin*, has also been made artificially by oxidising alizarin according to a special method.

Now I wish to speak, first, of the dyeing properties of these four colouring matters, all of which are manufactured on a large scale, and sold either separately or mixed together in various proportions, each maker giving his own peculiar brand or mark to the different qualities and mixtures. It is customary for the sake of simplicity to sell the colouring matters mentioned, whether they be separate or mixed, under the common name "alizarin."

Those which consist entirely or most largely of alizarin are called the "blue shades of alizarin," while those in which flavo-purpurin, or anthrapur-

purin predominate, constitute the "yellow shades of alizarin." These designations have arisen because the former dye alumina mordanted cloth a crimson or bluish shade of red, while the latter give a scarlet or yellow shade of red.

Commercial alizarin, numerous samples of which are shown on the table, is a light brown pasty mass of little or no value as a colouring matter if used by itself. If a piece of white calico or wool is boiled in water containing alizarin, it does not become dyed but merely stained a comparatively useless yellowish brown colour.

According to the old English authority on dyeing (Dr. Bancroft), colouring matters like alizarin which possess naturally no affinity for textile fibres, and cannot be fixed upon these without external aid, are called *adjective* colouring matters, while those requiring no such aid are termed *substantive* colouring matters.

I quite agree, however, with my esteemed friend Prof. Liechti, of the Vienna Dyeing School, that the time has now arrived when we must consider this classification as entirely obsolete, since it has neither scientific basis nor value.

It is well known that such a colouring matter as magenta, for example, may behave as a substantive colouring matter towards silk and wool, and as an adjective colouring matter towards cotton. The chemically different nature, indeed, of each individual colouring matter, and the different behaviour of the several textile fibres, compel us to find out the best method of fixing for each, taking into consideration, of course, class relationships; but at the same time these things prevent us from classifying colouring matters in the manner suggested by Bancroft, or from laying down any satisfactory theory of the fixation of colouring matters which is generally applicable.

From another point of view, however, we may distinguish two classes of colouring matters: (1) those which are themselves *colours* or pigments, and which need only to be indelibly and permanently fixed on the fibres in order that the latter may become dyed, e.g., indigo, magenta, etc.; (2) those in which the external colour has not the slightest relationship to the colours or coloured bodies obtainable from them in dyeing.

Now, alizarin belongs to this latter class, and may be well described as a *colouring principle*, capable of yielding a coloured body or pigment when it combines with some metallic oxide, e.g., oxide of iron, oxide of tin, oxide of aluminium, etc.

In these two vessels I have the oxides of two metals in a state of solution; in this there is oxide of iron, and in this oxide of aluminium. I will now pour into each a solution of alizarin, and we have at once coloured bodies produced in the form of insoluble precipitates or lakes. These coloured precipitates are compounds of alizarin with iron and aluminium respectively, and it is such insoluble precipitates as these, which constitute the colours produced during the dyeing with alizarin, upon wool or cotton previously mordanted with suitable metallic salts.

It is not always allowed that the coloured bodies or "lakes" fixed on the fibre are real chemical compounds, but although no sufficiently accurate analytical data have yet been obtained to prove this with absolute certainty, it seems to me it may well be conceded from the circumstantial evidence we have, e.g., in the case of alizarin red a *white* body alumina has united with a soluble *yellowish* substance alizarin, to produce an insoluble *red* coloured body; and if the alumina is replaced by other metallic oxides, other bodies possessing different colours are produced.

I may add that from what we know of the chemical constitution of alizarin, we may conclude that in



these compounds the hydrogen of the hydroxyl groups is replaced by the metal, and it is not at all improbable that when the constitution of the pure colouring principles of the various dyewoods comes to be determined, they will be found to contain similar hydroxyl groups.

If a dyer is asked what are the main qualities which he expects from any novel colouring matter presented to him by the colour manufacturer, his answer will probably be: "It must be cheap, easy of application, and fast." Now all these are characteristics of alizarin, and yet, strange to say, although it was discovered 16 years ago, I am afraid it is still almost a total stranger in most woollen dye-houses. How is this? Well, it is perhaps difficult to explain fully; but I trust you will excuse me if I give you one or two thoughts I have had regarding the probable cause. It has always appeared to me an unfortunate circumstance that the brilliant series of colouring matters first derived from aniline were so fugitive. There is an old adage—"Give a dog a bad name and it will stick to him." Now, as I have already said, I believe the deficiencies of these early aniline colours have been handed down to their successors without due discrimination, so that even at the present time, in the popular mind, the term "aniline" colour is almost synonymous with "fugitive" colour, and it is certainly generally regarded, though improperly so, as synonymous with "coal-tar colour." I am well aware that the requirements of some dyeing trades are not so exacting as those of others in respect of fastness and permanency of colour, so that even fugitive colouring matters have been much employed in them. Indeed it is difficult to say how great may have been the influence for good of this demand, in keeping alive the manufacture of even fugitive colouring matters and thus stimulating that particular field of chemical research which has at length yielded colouring matters of greater permanency.

Now, although alizarin is not an aniline colour, it would, owing to its origin from coal-tar, probably be considered so by many, and not be credited with that superlative quality of permanence and fastness which it really possesses.

Perhaps a more potent reason for the neglect which alizarin has experienced at the hands of the woollen dyer resides in the fact already stated that it requires the aid of a mordant, not only to fix it on the fibre, but also to develop its colouring power. I am inclined to think that the excessive simplicity of the methods of applying the great majority of the coal-tar colours has to a considerable extent spoiled many dyers, so that they are apt to take less notice of a new colouring matter if it offers even slight difficulties in this respect.

Quite naturally they are anxious that it should require only one bath, perhaps that it should dye in an acid bath, that it should dye level without requiring care in the manner of heating the dyebath, and so on. All these may be very desirable, but unfortunately alizarin does none of these things. But why should a valuable colouring matter suffer neglect because of these or similar reasons? The old dyewoods, *e.g.*, logwood, madder, cochineal, camwood, &c, also require two baths, and they are general favourites. So sensible, indeed, have the manufacturers of coal-tar colours become of this liability of novel colouring matters to neglect, on the part of dyers, that they employ special chemists who experiment with all newly-discovered dyes before they are issued to the trade. You all know the well-arranged pattern cards (several of which are exhibited on the table) which are ever and anon distributed by various coal-tar colour manufacturers, and yet one sometimes hears the remark

that the receipts which accompany them are not worth much, and may even be misleading.

If this be really so (though I am by no means inclined to be so adversely critical), then I say there is every reason why such an abnormal state of things should cease.

I take it that the province of the colour manufacturer is rather to *make* colouring matters, than to say how they can be best applied. Indeed, although it would be very useful and is even desirable, it is absolutely impossible for him to be conversant with all the varied and special requirements inherent to each particular dyeing trade, and hence he can only give indications as to possible methods of application and at most general instructions. On the other hand, it is the bounden duty of every dyer to be possessed of that knowledge which will enable him to determine the best method of applying each or any colouring matter to his own particular style of goods.

The necessary experiments, however, require considerable care, time, and attention, if they are to lead to any useful issue, and these factors are seldom at the disposal of a dyer busily engaged in the oversight of his business.

The natural conclusion, therefore, is (and I would seriously urge it upon your attention), that every dye-house should, if possible, have its competent chemist or colourist, with properly equipped laboratory, who should, among other things, devote himself to a careful study of the application of the various colouring matters to the special requirements of the trade of the firm with which he is connected, and who should work hand-in-hand with the dyer, endeavouring to improve old processes, and to discover new ones. In the present age of keen competition and rapid interchange of ideas, you are aware that he who introduces some novelty in colour, style, or effect, etc., has the best chance in the race, and by such division of labour as I have alluded to, more rapid progress in this respect becomes possible. To return to my point, I believe the present meagre employment of alizarin by woollen dyers is in a large measure owing to the fact that it has not become the general custom with them to experiment largely with the novel dyes, nor to employ chemists in the manner I have mentioned. Quite a different state of matters has reigned among calico printers. Most of them, especially large firms, have for a long time recognised the value of chemists, engaged as above described; and even when the price of alizarin with 10 per cent. of solid matter was 10s. per lb., or nearly twenty times its present price, and the product was far from being so pure and regular as it is now, these printwork-chemists were busily experimenting with it in a thousand ways. The result has been that in the calico printing and Turkey red dyeing trades alizarin has entirely displaced its old rival madder, and is the principal colouring matter employed. No doubt alizarin has properties which render it of very special importance to the trades mentioned; but still its *rapid* introduction has in no small degree been owing to the busy army of colourists or chemists hard at work on it in the various calico-print works throughout the length and breadth of Europe.

Having spoken then of some of the possible hindrances which may have stood in the way of the adoption of alizarin by woollen-dyers, let me now enumerate its chief characteristics of value which ought at once to secure its universal employment by woollen-dyers, especially those engaged in the heavy-woollen, tweed, carpet, shirting-trade, &c., that is wherever the dyed goods are subsequently scoured or milled or have to bear constant exposure to light.

In the first place it is a cheap colouring matter; an alizarin paste containing 20% solid matter costs at the present time about 1s. 4d. per lb.

Then, secondly, the colours it yields leave little or nothing to be desired with respect to fastness, to scouring, milling, light and wear. In these respects it may be regarded as a worthy colleague of indigo, and deserving of equal attention.

Thirdly, it is capable of giving, even when used alone, *i.e.*, without admixture of other dyestuffs, a large variety of colours, by simply varying the mordant, *e.g.* red, orange, lilac, black, brown, etc.

Being convinced that alizarin ought to occupy a much better position among woollen-dyers than it does at present, and believing, too, that it *must* hold, ere long, a *very prominent position*, so soon as its excellent qualities are fully and generally recognised, I thought it would interest the members of this Society if I brought before them the results of a few experiments relative to its application in wool dyeing made by some of the students of the Yorkshire College, Leeds, during the past session, and I am happy to state that many of the experiments to which I shall refer, have been carried out by Mr. W. Ray, a former student of our distinguished chairman.

Now the problem which the dyer has to solve is: Under what conditions can these coloured precipitates be produced upon the fibre so that when dyed it shall exhibit the fullest development of colour with the least expenditure of alizarin, together with the greatest brilliancy, fastness and evenness of shade, and with the least injury to the textile material, etc.?

A wide field of experiment is here open to the dyer, and I propose to explain what system he may adopt in order to determine how alizarin is to be applied in wool dyeing.

But first as to the mode of experiment. In order to determine the effect of each particular ingredient used, the student must perform simultaneously two or more distinct experiments in which equal weights of the same textile material are submitted to all the necessary operations under precisely the same conditions, except as regards the amount employed of the ingredient whose action is to be studied. Whatever indeed be the factor the influence of which is to be determined, whether it be the duration or temperature of mordanting or dyeing, the character or amount of the several ingredients employed, and so on, *that factor alone* is varied while the others remain unchanged. In this way a systematic series of dyeing experiments is carried out; one by one the nature and value of each individual influence is carefully determined, until at length by a cumulative process the *totality of conditions* necessary to produce the best results is accurately determined. The actual number of experiments which it may be requisite to carry out with any given colouring matter, before arriving at a full knowledge of its dyeing properties, is quite indefinite, and is more or less determined by the character of the colouring matter, and by the general and special chemical information of the experimenter.

Now, as to the apparatus required, it is comparatively simple. A water or steam-bath, or an oil-bath heated with gas or steam, provided with a perforated cover for the reception of the dye-vessels, serves for the simultaneous and equable heating of the latter. The dye-vessels themselves should be of hardened glass or well glazed porcelain or earthenware, and capable of holding from 1 to 2 litres. Metallic vessels of whatever kind, although very useful for certain classes of work, are not to be recommended for general use in experimental wool-dyeing, since the acids and acid salts often used in mordanting and dyeing, invariably dissolve traces of the metal, which

in many cases affect the ultimate result. For the same reason stirring rods or other apparatus for moving the textile material during the dyeing process, must also be of glass or porcelain. A good balance, a few glass beakers, porcelain basins, measure glasses, burettes, pipettes, and hydrometers, complete the equipment.

The first series of dyed patterns on the diagram were produced with the object of determining approximately what percentage of aluminium sulphate wool should be mordanted with in order to give the best red when dyed subsequently with alizarin.

Six pieces of woollen cloth were mordanted with varying percentages of aluminium sulphate, the cloth being immersed in the cold solution, which was then very gradually in the course of one hour heated to 100° C. and boiled for half an hour. After washing well, they were dyed in separate baths with equal weights of alizarin.

This experiment certainly showed that wool is capable of decomposing aluminium sulphate, and takes up certain of its constituents when boiled with its solution, and in so far it illustrates the general principle upon which the ordinary method of mordanting wool is founded, but it also showed that the use of aluminium sulphate *alone* in the mordanting bath, is insufficient to fix either the necessary amount or the proper form of alumina in the fibre, since none of the colours produced were remarkable either for depth or brilliancy. Now general experience leads one to expect that an addition of cream of tartar may be beneficial.

A second experiment, therefore, was carried out to determine the best *relative proportions* in which aluminium sulphate and cream of tartar should be used. For this purpose six pieces of woollen cloth were now mordanted with 6% aluminium sulphate and cream of tartar varying in amount from 0% to 85%. The subsequent dyeing with alizarin was of course the same for each piece of cloth.

This experiment showed that the use of cream of tartar along with aluminium sulphate in the mordanting bath is all important, there being a marked improvement in colour with the addition of even a small amount.

The best relative proportion of cream of tartar to employ with 6% aluminium sulphate seemed to be from 3.5–5%. These amounts represent 1 mol. aluminium sulphate, and 2 to 3 mols. cream of tartar; it would appear therefore as if no exact chemical relationship between the two were absolutely necessary in order to ensure the best result.

A third experiment was now made for the purpose of determining the best *absolute amounts* of aluminium sulphate and cream of tartar to employ. In this trial the amounts of aluminium sulphate used varied from 2 to 12 per cent., the cream of tartar varying in proportion according to the result of the previous experiment. The result now obtained was, that when dyeing a red with 10 per cent. alizarin (containing 20 per cent. solid matter), woollen cloth should be mordanted with such proportions of materials as fall within the following limits: 6 per cent. aluminium sulphate + 3.5 to 5 per cent. cream of tartar and 10 per cent. aluminium sulphate + 5.9 to 8.5 per cent. cream of tartar; or as the medium amount, say 8 per cent. aluminium sulphate + 7 per cent. cream of tartar. If a *deficiency* of aluminium sulphate be employed, a poor dull colour is produced, with *excess*, the colour although bright, is deficient in intensity. Addition of too little tartar gives a poor lifeless colour; excess is less injurious. It is well to state here, that the weight of each piece of wool dyed, was 10 grms., and that the mordanting and dye-bath contained one litre of distilled water. The percentages



refer to the weight of wool employed. As to the use of other aluminium salts experiment shows that ordinary alum may be employed in the same way, and with good results. In a discussion on a paper of which this may be considered an extended edition read recently before the Society of Dyers and Colourists at Bradford, Mr. Leviustein suggested that the use of the sulphocyanides of aluminium and chromium might give good results in wool dyeing. Accordingly, having made experiments recently in this direction, I may now state that aluminium sulphocyanide is capable of giving excellent results without the necessity of adding cream of tartar along with it, and interesting questions to which time will not permit me to refer are thus raised.

I pass now to consider the dyeing operation.

One of the most interesting points in connection with the employment of alizarin in dyeing, is the necessity of using a lime salt in the dyebath.

Already a century ago it was recognised that certain varieties of madder (*e.g.*, Alsace madder) which contained little or no lime, required the use of a calcareous water in the dyebath. It is related that a Turkey red dyer, Hausmann, who had produced good reds in Rouen, was unable to do so when he removed his business to a place near Colmar. Possessed, however, of a good chemical knowledge, Hausmann discovered that the water supplied to his new works contained no lime, as did the Rouen water, and having the idea that madder contained some acid body which prevented its colouring matter from combining with the mordant in the cloth, he thought that it was the bicarbonate of lime present in the Rouen water which acted beneficially by neutralising this acid of the madder without affecting the colouring matter. Hausmann reasoned therefore that if he added a certain proportion of carbonate of lime or chalk to the water of the dyebaths in his new works, he ought to obtain good Turkey reds as before. His theoretical considerations seemed to receive confirmation by the success which attended the practical application of this idea.

Since that day, other explanations of the necessity of adding chalk or some other lime salt to the madder dyebath, have been advanced, since the addition has been found to be equally necessary in the case even of chemically pure alizarin.

It has been noticed by Schlumberger, Köchlin and others, that certain colours produced by the combination of the colouring matter with two mordants are faster than when a single mordant has been employed. It would appear, according to Schlumberger, that alumina mordanted cotton cloth takes up in the madder dyebath lime as well as alizarin, and thus gives rise to an alumina-lime-alizarin lake, which is faster to boiling soap than a simple alumina-alizarin lake. An analysis of the red pigment on madder dyed cloth has shown that it contains two molecules alumina ( $\text{Al}_2\text{O}_3$ ) and three molecules lime ( $\text{CaO}$ ).

When a calcareous water is used in dyeing, a lime-alizarin lake is formed, which although insoluble in water, is soluble in the presence of free carbonic acid, the more so as the lime is present mostly as bicarbonate. This dissolved lime-alizarin lake is said to combine with the alumina on the fibre, so that the ultimate pigment or colour produced, is a triple compound.

A modern explanation therefore of the point in question is, that when the water is not calcareous, it is necessary to add to the dye-bath a lime salt in order to produce the above mentioned soluble lime-alizarin-lake. In cotton dyeing, chalk is usually employed, in the proportion of two per cent. of the weight of an alizarin containing 20 per cent. solid matter. This

chalk is for the most part dissolved by reason of the free carbonic acid in the water.

Now during the gradual raising of the temperature of the dyebath, carbonic acid is driven out, and there is always the possibility of insoluble lime-alizarin-lake being thrown down and a certain proportion of the alizarin becoming thus inert and lost. Hence Rosenstiehl has recommended the use of acetate of lime instead of the carbonate, or, in the case of highly calcareous waters, he recommends them to be neutralised with acetic acid.

In this case the alizarin dissolves at first in the acetate of lime, and then, as the temperature rises, decomposes it to form a lime-alizarin-lake, which then remains dissolved in the liberated acetic acid, and gradually combines with the alumina on the fibre.

A comparative dye trial showed that when wool has been properly mordanted with aluminium sulphate and cream of tartar, the dyebath should contain for 10 per cent alizarin (containing 20 per cent. solid matter), four to six per cent. solid acetate of lime (91.8% strength), or 40 to 60 per cent. of the weight of alizarin employed. That, in order to obtain a good result, one should use in wool dyeing 30 times as much acetate of lime as in cotton dyeing is somewhat remarkable. Rosenstiehl has shown that aluminium mordants on cotton are properly saturated if two molecules of lime are used for each molecule of alizarin. The amounts actually used in the above experiment represent 3 to  $4\frac{1}{2}$  molecules calcium acetate to one molecule alizarin. Without the use of acetate of lime, only a worthless pink colour is produced, with excess the red becomes darker, but lacks brilliancy. The maximum brilliancy of colour is obtained when care is taken that the calcium acetate is in nowise alkaline, viz., by neutralizing or even slightly acidifying it with acetic acid. As might be expected from this, the oxide and carbonate give less brilliant reds. If calcium acetate be replaced by the acetates of K, Na,  $\text{NH}_4$  and Mg, the dyeing power of the alizarin seems to be fully developed, but the red is of a decided crimson hue, and possesses by no means the brilliancy of a good standard red. Barium acetate gives a similar result. Of all the acetates which have been experimented upon in the Yorkshire College dye-house, that of strontium gives a result most nearly approaching that given by calcium acetate, the red produced being a bright scarlet.

Having determined the proper amount and proportion of ingredients to employ both in the mordanting and in the dye-bath, it remains to determine under what conditions of temperature the mordanting and dyeing should take place.

Experiment shows that when mordanting with aluminium sulphate and cream of tartar, full and level colours only result when the textile material is entered into the bath at a low temperature and this is gradually raised to  $100^\circ\text{C}$ . and the boiling is continued for half an hour or more.

As to the method of dyeing, the same precautions must be adopted. It is quite possible to exhaust the dyebath at temperatures below  $100^\circ\text{C}$ ., *e.g.*, at  $80^\circ\text{C}$ ., but the red is then devoid of brilliancy. Comparing the shades of colour given on alumina mordanted wool, by the various members of the alizarin series, it will be seen that alizarin and purpurin both give a deep bluish-red or claret-red colour, while iso-purpurin and flavo-purpurin give bright reds.

By pursuing a similar systematic course of dye trials with stannous chloride as the mordant, the following results have been arrived at.

The proportion of mordant found to give the fullest colour is six to eight per cent. stannous

chloride (tin crystals) with equal weights of cream of tartar. The use of acetate of lime in the dyebath in this case is not absolutely necessary.

*Without* acetate of lime, alizarin produces a good bright orange; *with the addition*, the colour becomes redder, quite approaching a good scarlet. Acetate of lime may be added to the dyebath to the extent of even 30 per cent. (that is, 300% of the weight of the alizarin employed), and yet the colour remains fairly bright, but redder than when less amounts are used. Stannous chloride mordant always renders wool harsh to the feel, and excess materially destroys its milling property, so that, although 6 to 8 per cent. stannous chloride (cryst.) may give the fullest colour, it is scarcely advisable to employ in practice more than 4 to 5 per cent. stannous chloride, with, of course, an equal amount of cream of tartar.

On comparing the colours given by the different members of the alizarin series with stannous chloride mordant, and *without* acetate of lime in the dyebath, it is seen that those yielded by alizarin, iso-purpurin, and flavo-purpurin form a graduated series of bright oranges, that given by alizarin being the reddest, and that given by flavo-purpurin the yellowest in tone. Their brilliancy approaches that of the azo-oranges. The colour yielded by purpurin is very different, being a dull red colour. The addition of acetate of lime to the dyebath makes all the colours redder and less susceptible to the action of milling, which otherwise always makes them much redder.

Referring now to the use of *bichromate of potash* as a mordant for alizarin, experiment shows that the most suitable amount to use is 3 per cent., with the addition of 1 per cent. sulphuric acid at 168° Tw. Very much larger proportions may be used, e.g., 10 per cent. bichromate of potash, and 20 per cent. sulphuric acid; these are, however, by no means to be recommended on economical grounds, but it is interesting to note the fact that not only is the colour not destroyed, as in the case of some colouring matters, but it is actually darker than with the smaller amounts. The addition of the 1 per cent. sulphuric acid to 3 per cent. bichromate of potash adds decided intensity to the colour. A very interesting fact in connection with the use of this mordant in alizarin dyeing, is, that the addition of acetate of lime to the dyebath is not only not essential to the production of a good full colour, but unlike even the case of using stannous chloride, its addition causes very little difference of colour, simply making it slightly bluer in tone. If patterns in which aluminium sulphate is the mordant employed, are compared with corresponding patterns in which bichromate of potash is used, the great difference in the action of the acetate of lime in the two cases is at once apparent.

Being struck with this result, I was anxious to see if the use of acetate of lime was equally unnecessary when other chromium mordants were employed. Experiment shows, however, that when wool is mordanted with chrome-alum and tartar, the use of acetate of lime is just as necessary to the production of a full colour as in the case of using aluminium sulphate mordant. It would appear then that the particular state in which the mordant is fixed upon the cloth, is not without its influence in determining the necessity or otherwise, of using acetate of lime in the dyebath. I am inclined to think that when wool is mordanted with 1 mol. bichromate of potash and 1 mol. sulphuric acid, which the proportions recommended above actually represent, or practically with chromic acid, there is fixed upon the fibre simply chromic oxide, and this may at once combine directly with alizarin to form a chrome-alizarin lake. Experiment shows indeed that if the mixture of  $K_2Cr_2O_7$  and  $H_2SO_4$  be substituted by  $H_2CrO_4$ , equally good results are obtained.

In the case of mordanting with chromium and aluminium sulphates it is generally assumed that basic sulphates of these metals are deposited on the fibre. If this be so, may not then the dyeing with a solution of a lime-alizarin-lake be necessary in order that double decomposition may take place between the basic sulphate and the lime-alizarin-lake, producing calcium sulphate and aluminium- or chromium-alizarin lake? This supposition would, of course, exclude the idea mentioned above that lime is a *necessary* constituent of the red or chocolate coloured lake.

It is, however, useless to speculate without the aid of further experiments, and these I hope to carry out.

Alizarin, iso-purpurin, and flavo-purpurin give with bichromate of potash mordant, a series of claret browns, alizarin giving the bluest and darkest colour, flavo-purpurin the reddest and lightest. Purpurin gives a very much deeper brown even than alizarin, and seems to be the best colouring matter to employ with bichromate of potash mordant.

With *iron mordants* alizarin and its allies give very fast but not bright shades of lilac and purple. Experiments have been made by my students for the purpose of comparing the different effects produced by various ferrous and ferric salts, but I must leave any notice of the results obtained to some future time, since the experiments are far from being completed.

To obtain the best and fullest shades, I may say that what dyers consider abnormally large percentages of cream of tartar are found necessary.

*Nitro-alizarin*.—This colouring matter, generally called "alizarin orange" is produced by the action of nitrous acid on alizarin. With *aluminium* mordant it gives an orange colour, which is fast to light and milling. With *bichromate of potash* mordant it gives a light brown colour. With *stannous chloride* mordant it gives also pale brown colours, which are rendered darker if acetate of lime is added to the dyebath. It is quite evident, however, that this mordant destroys the colour more or less, probably because of its reducing action. With *stannic chloride* better results are obtained, quite a good orange red being produced. With *iron mordant* it yields a good claret-brown colour.

*Alizarin Blue*.—This colouring matter is produced from nitro-alizarin by the action of sulphuric acid and glycerine at a high temperature. It comes into the market in two forms, viz., as alizarin blue paste (containing 10 per cent. dry substance or less), and as alizarin blue S in the form of a dark powder. The former is insoluble or very difficultly soluble in water, while the latter is soluble.

Alizarin blue S is really a compound of bisulphite of soda with the insoluble form of alizarin blue, and it is well to remember that its solutions decompose, if heated to 70° C., with precipitation of the insoluble form of blue. When, therefore, alizarin blue S is used, the dyeing must be mainly completed below this temperature. With lime it forms an insoluble lake, hence the presence of lime salts in the dye-bath must be avoided, otherwise there will be a loss of colouring matter.

The insoluble form of alizarin blue may be applied in dyeing according to the indigo vat method, by reducing it with zinc powder and carbonate of soda, or by the method of mordanting and dyeing in separate baths. When the latter method is employed one may either add to the dye-bath a certain proportion of bisulphite of soda to render it soluble, or the dyeing at 100° C. must be long continued.

The following notes refer only to the use of alizarin blue S, but my experiments with it are far from complete.



The most suitable mordant to use is *bichromate of potash* in the proportion of 3 per cent. of the weight of wool. The addition of sulphuric acid is not beneficial, but if used at all, its amount must not exceed 1 per cent., otherwise the colour is rendered dull. Good colours are also obtained by using cream of tartar instead of sulphuric acid.

In order to be quite sure of obtaining level shades, the method of dyeing employed in my experiments has been to work the woollen cloth for half an hour in the cold, then to heat the bath gradually to 60° C. in the course of half an hour, and to dye at this temperature for one hour; the temperature was finally raised to 100° C. in the course of another half hour, and the boiling continued for half an hour longer. The addition of acetate of lime to the dye-bath makes the colour dull and less intense, even though it be added only towards the end of the dyeing operation. The addition of chalk to the dye-bath is much more injurious than acetate of lime.

The colour obtained by using five per cent. alizarin blue S is a medium indigo blue shade. This colouring matter is indeed the most formidable rival of indigo which has yet appeared, the colour it yields being equally fast to scouring, milling, light, &c., and it has the decided advantage of not rubbing off.

One other characteristic of this colour which may be thought worthy of mention, is that it shows a moderately good imitation of the indigo nitric-acid test, *i.e.*, when spotted with strong nitric acid the colour becomes more or less yellowish, which, however, eventually changes to a reddish brown. I don't know that this fact is of any real significance, but it may possibly induce some merchants to take more kindly to its introduction than they might otherwise be disposed to do.

When *aluminium sulphate* is the mordant employed a purplish-blue colour is obtained. The best proportion seems to be six to eight per cent. aluminium sulphate with the addition of five to seven per cent. cream of tartar. The addition of acetate of lime to the dye-bath is not beneficial, it makes the colour bluer and less intense.

With *aluminium mordant* the colour seems to have a great tendency to become uneven unless very particular care is taken in mordanting, and the wool has always a somewhat harsher feel.

With *stannous chloride* as the mordant alizarin blue S gives quite a purple colour, much redder even than when an aluminium mordant is employed. Use four per cent. stannous chloride (crystals) and two per cent. cream of tartar. Stannous mordants do not appear to be altogether suitable for employing alone, not only on account of the harshness imparted to the wool, but because the dyebath always contains a certain amount of precipitated colouring matter.

*Ferrous sulphate* as a mordant for alizarin blue S is also little suitable. It gives a greenish blue colour, having little brilliancy, and apt to be uneven. Mordant with four per cent. ferrous sulphate, and eight per cent. cream of tartar.

*Gallein*.—This colouring matter is produced by heating to a high temperature a mixture of phthalic anhydride, and pyrogallol. It comes into the market as a dark brownish red paste, containing 10 per cent. solid matter, sometimes less. In dyeing properties it somewhat resembles haematein, the colouring matter of logwood, and it certainly deserves the attention of woollen dyers.

With *bichromate of potash* as the mordant it gives a moderately bright purple colour. The best amount to employ seems to be two per cent. bichromate of potash; it is quite imperative that no addition of sulphuric acid be added to the bath, otherwise, the colour is rendered very much duller, as it is also with

the use of excess of bichromate of potash. In these respects it differs materially from haematein.

With *aluminium mordant* a much redder purple is obtained than with chromium mordant. Use six per cent. aluminium sulphate and five per cent. cream of tartar. The addition of one to two per cent. acetate of lime to the dye bath adds a little brilliancy and intensity to the colour. The addition of chalk to the dye bath is not advisable; even with two per cent. of chalk, the colour produced is considerably weaker.

With an *iron mordant* gallein gives a deep violet colour. Use eight per cent. ferrous sulphate and five per cent. cream of tartar.

There seems no particular difficulty in obtaining level colours with gallein, but it is always advisable to enter the wool into the dye-bath at a low temperature, and to raise the temperature *gradually* to the boiling point. With 10 per cent. gallein paste, containing 10 per cent. solid matter, colours of medium intensity are obtained.

*Cerulein*.—This green colouring matter is produced by the action of sulphuric acid on gallein at high temperatures.

Like alizarin blue it comes into the market in two forms, soluble and insoluble: the former is a compound of cerulein with bisulphite of soda, and is sold either in the form of a greenish black powder as cerulein S, or as a paste.

The most satisfactory results with regard to evenness of shade are obtained by using the soluble modification, and the same mode of heating the dye-bath as adopted in the case of alizarin blue S is desirable, and for the same reasons.

All the colours given by cerulein with the usual mordants of chromium, aluminium, tin and iron, are exceedingly fast to light and to milling, and it seems to me that in these respects it could well substitute certain shades of indigo-green, *e.g.*, combinations of vat indigo with old fustic yellow.

Much less variety of colour is given by cerulein when used with the different mordants than is the case with alizarin. All mordants simply cause it to dye certain shades of dark green of an olive or myrtle tone.

The most serviceable mordant seems to be bichromate of potash. Mordant with two per cent. bichromate of potash, with the addition of 0.7 per cent. sulphuric acid at 168° Tw. The addition of chalk or acetate of lime to the dye-bath is to be avoided. By dyeing with cerulein S powder varying in amount from 1.5 per cent. to 10 per cent., a nice range of colours, from pale sage-grey to dark indigo green, is obtained. Comparing this latter with an approved shade of vat-indigo-green it is very similar to it when looked down upon, but slightly more olive or less blue when examined overhead. All the colours can readily be obtained level.

When an aluminium mordant is used the green has a more greyish tint, and there is a great tendency to uneven dyeing.

With stannic chloride as the mordant, cerulein S gives greyish blue-green colours, the pale shades of which promise to be serviceable. Mordant with such an amount of stannic chloride equivalent to five per cent. stannous chloride (crystals). With the addition of cream of tartar to the mordanting bath the normal bluish-green colour is obtained, but the amount of tartar to be added for this effect is prohibitory. With iron mordants dark olive-greens are obtained.

Although in the above reference has been made to the method of determining the conditions of dyeing, etc., necessary to obtain the brightest and most intense colours, it must not be forgotten that a dyer knows but half his business if he is simply acquainted with these conditions. He must not only know how

best to dye any given colour : he must also know the capabilities of each dyed colour : how it withstands the action of light, milling, scouring, etc.,—in short all those influences, whether natural or artificial, to which the dyed fabric is likely to be submitted. Hence all dyers should habitually and systematically expose portions of dyed patterns to the several influences above-mentioned, all care being taken to avoid the possibility of referring any effect produced to more than one cause at a time. Such exposed patterns must be afterwards carefully compared with the original patterns as dyed.

In conclusion, I may say that with the combined use of two or more of the colouring matters spoken of, in conjunction with one or more judiciously selected mordants, it has now become possible to the dyer to obtain an indefinite series of *fast shades* of brown, drab, olive, grey, etc.; indeed, all those tertiary shades for the production of which he felt it necessary formerly to have recourse to the dyewoods. There is, however, nothing to prevent him using extracts of the dyewoods *along with* these colouring matters should he find this necessary to the production of some special shade of colour. But the compound shades obtained from these colouring matters of the alizarin series, are decidedly faster to light and other influences than those generally produced by means of the dyewoods. In the production of these shades the dyer makes use of such colouring matters as will give, when employed separately, red, yellow, and blue colours, or their equivalents, *e.g.*, orange and blue, red and green, purple and yellow. It is further necessary, or at least very desirable, that all the colouring matters employed should be applicable by the same process, so that they may be added to the same dyebath. Finally, it is important that the constituent colours (red, yellow, blue, etc.) should be equally fast. In short, the dyeing properties of the colouring matters employed, and the physical and other properties of the colours obtainable from each, should be as similar as possible, if the best class of compound shades are required. Now, when we consider what choice of fast red, yellow, and blue colouring matters there is among the dyewoods, we find that for the fastest red we must use madder, for yellow—weld, and for blue—indigo. I refer here only to the fastest colouring matters we can employ. It is at once evident to the dyer that, in using this combination, the indigo must be applied separately, *viz.* : by the "vat method," hence with possible reluctance he generally uses, instead of indigo, the more conveniently applicable but more fugitive logwood. For other reasons, weld is replaced by old fustic, and madder is more or less substituted by camwood and other red dyewoods. The result of these various changes is, that the compound shades thus obtained fall more or less from the high standard of fastness and permanency attainable even with the dyewoods.

Now, although the dyeing properties and the physical characteristics of, the colours yielded by the alizarin series of colouring matters including the various "alizarins" alizarin blue, cerulein, and even gallein, are not as similar for the purpose under consideration as could be desired, still they are sufficiently alike to render their use, for the purpose of producing fast compound shades, a distinct advance on that of the old dyewoods.

Time will not permit me to discuss at length the pros and cons of the modern fast coal-tar colouring matters and our time-honoured dyewoods. At present there are some of the latter which the dyer could very ill spare, and there are gaps to be filled up in the ranks of the former, but I feel assured of this, that sooner or later the dyer must make up his mind

to bid a long and last farewell to his old and valued friends, and so I would urge each one to learn, by some such method as I have indicated, the capabilities and characteristics of these new and interesting acquaintances.

#### DISCUSSION.

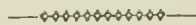
The CHAIRMAN : You have now heard what is done in the Dyeing School of the Yorkshire College, and how the new processes of dyeing woollens are systematically worked out. It has shown me what before was not very clear—how the dyeing processes are taught in the dyeing schools, and I quite believe that work of this kind is not only of practical value, but has an educational influence.

Dr. HEWITT : I cannot speak as a calico printer or dyer, but it struck me that Mr. Hummel's paper was a wonderful answer to the Chairman's suggestions as to the importance of technical instruction. We shall have to put away the old rule-of-thumb methods and adopt strictly systematic methods, which would lead to useful results, Sir Henry had referred to methods which, he thought, should be adopted for promoting and assisting technical research. In a discussion which took place in the Liverpool section, a short time since, there was an expression of opinion that the same course of instruction could not be useful to both masters and men, and the degree to which, after all, either the one class or the other could obtain definite technical knowledge was a question of money, money, money; but there could be little doubt that to fit a man to control a large manufactory his education in science could not be too widely based, as it is not merely chemistry, but physics, engineering, and drawing are all aids to the practical man in managing a works.

Mr. KNOWLES : I rise with great pleasure to second the vote of thanks. If we in Lancashire are to maintain our ground and be prosperous in future competition with the chemistry of Germany and France we must be up and doing. I am glad to see that the desire for this particular knowledge is gaining ground, and what this college is doing will enhance the well-being of this great commercial community. Mr. Hummel has said that the blue he referred to would dye woollen goods as fast as indigo, and would stand the light and soaping quite as well. I should like to know if the same remark applies to cotton goods?

Mr. HUMMEL : My experiments with this colouring matter hitherto have chiefly had reference to its applicability to the woollen fibre. I have not yet exposed to the light alizarin blue dyed cotton, and prefer therefore to allow those who have to give an authoritative answer to the question.

Mr. C. O'NEILL : Alizarin blue when properly applied upon cotton goods is as fast as indigo blue to the action of soaping and of light.



#### THE EARLIEST RECORDS OF METHODS FOR THE COKING OF COAL IN COKE OVENS FOR METALLURGICAL PURPOSES, WITH RECOVERY OF THE TAR AND AMMONIA.

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It would appear from testimony, which I received in the shape of a letter on October 21st, from Dr. Adolf Gurlt, of Bonn, author of a work tolerably well known in Germany, *Die Bereitung der Steinkohlenbriquettes*, that as early as the year 1764, the thought seems to have occurred to a certain manager of coke ovens



near Saarbrücken, to mitigate the dense smoke evolved from his short coke-oven chimneys, by collecting the tarry and nitrogenous matters (ammonia), otherwise lost. The thought seems not only to have occurred, but to have been developed into a plan which was actually carried out. The only unfortunate circumstance for the individual in question, to whose genius and enterprise the process of recovery was due, is that at that date chemical science was in its earliest infancy—it was little more than cradled, if I may be allowed the expression—and so the old philosopher, to be soon mentioned by name, appears to have been animated in his enterprise by what was little short of the spirit of prophecy, in seeking to recover from his coke-ovens substances, which at that early period were of very limited application, and of little monetary value, though now, more than a century later, in one of them (coal-tar) we have the source of the brilliant coal-tar colours, so long after destined to become a very considerable source of the revenue of the *Vaterland* of this old pioneer, and in the other (the ammonia) we have the essential part of the manurial compound, ammonium sulphate, so extensively employed for a chief branch of agriculture in Germany—the growth of the sugar-beet.

In the work of De Gensanne, "*Traité de la fonte des Mines par le feu du Charbon de Terre*," two vols., 4to. Paris, 1775 and 1776, is to be found (Vol. i., page 263 *et seq.*, and in Tables 32, 32) the description of singular coke ovens formed of dome-like fireclay retorts, which were heated from the outside, and

visit to the alum works and the "burning hill" of Dutweiler near Saarbrücken. Here he met with an old technologist named Stauf, who inhabited a lonely cottage in the forest, and this Herr Stauf, Goethe called a "Kohlenphilosoph," and a "Philosophus per ignem," and described him further as a worn-out, haggard little man, with a boot on one foot and a slipper on the other. Stauf was the manager of the works, and he conducted the visitors up the hill to a row of coke ovens, where coal was "desulphurised" for the use of iron works, and where old Stauf had produced oil, "bitumen," and lamp-black. Moreover, not ignorant apparently of the value of ammonia, he had also made a cake of sal ammoniac, collected from the "burning hill." The works, however, did not seem to pay, and the old philosopher feared they would have to be abandoned.

It is extremely probable, as Dr. Gurlt thinks, that both localities described by De Gensanne and by Goethe are identical, for Sulzbach, mentioned by the former, and Dutweiler (now Dudweiler on the map), mentioned by Goethe, are only a couple of English miles apart. Although De Gensanne (in 1764 or 1765) makes no mention of the quaint old philosopher Stauf, whom Goethe saw in 1771, he may still none the less have been the originator of the ovens at Sulzbach, and most certainly, as Gurlt says "his name ought to be preserved in the annals of chemical technology."

From the description of the construction of Stauf's ovens given by De Gensanne (assuming the plant

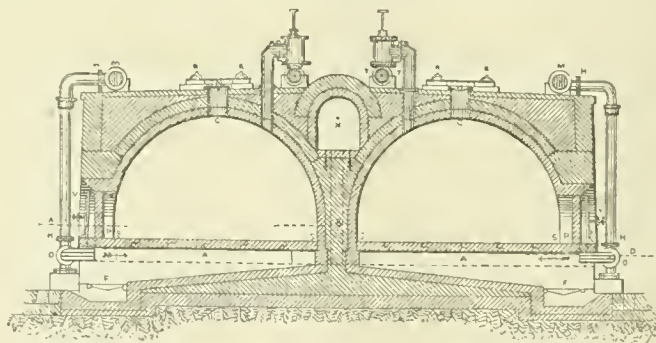


FIG. 1.

which before the year 1767 were in actual operation at the ironworks (blast furnaces) of Sulzbach, near Saarbrücken (probably about 1764), and were visited by De Gensanne. There were nine ovens side by side in a battery, of which three in a group were always in the same stage of distillation, and each oven required three days to work off its charge, so that each group of three worked off three charges in this space of time. These retorts, or rather "muffles," were charged by a door at the front, and at the back was a pipe made of copper, which formed the continuation of the inclined bottom. Through this pipe the liquid products escaped, "les huiles et bitumes," and collected in a large pot with a lid, a kind of tar-well. According to De Gensanne the "bitumen" sank to the bottom, whilst the "oil" resembled distilled petroleum (page 277), though less inflammable, and was used for burning in peasants' lamps, and in the miners' lamps in the Sulzbach coal mines; "its smell was bituminous, and it smoked much."

Now, in 1771, Goethe, then a student of Strassburg University, made a trip on horseback, accompanied by his friends Eglebach and Weyland, through Lothringen and the principality of Nassau-Saarbrücken. In his "*Aus meinem Leben*," Wahrheit und Dichtung, Part ii., book x., Goethe describes his

described by De Gensanne and by Goethe to be identical), it is pretty certain that he is right, and that the oils would resemble petroleum oils, and would certainly contain paraffins, for the mode of applying the heat to the coal in ovens of such a shape (dome or muffle-shape), and from the fact that the coal must have lain in dense masses on the oven floors or beds, the charges being large ones requiring "three days to work off" would preclude the possibility of obtaining other than what might be termed the lower temperature products, in the shape of tars, *i.e.*, tars of lower specific gravity, and resembling in composition shale-tars.

Stauf's oven would, in general principle, bear a rude resemblance as regards mode of applying the heat, charging the fuel, &c., to the more elaborate and modern Pernolet's adapted Beehive (Fig. 1), and just as the latter oven yielded a tar of low specific gravity under the special conditions of carbonizing, so in all probability Stauf's coke-oven-tar would be one of low specific gravity, and contain abundantly bodies at present characterizing shale-tars.

Most of the German text-books of technology have quoted Gurlt, where in his work, already mentioned, *Die Bereitung der Steinkohlenbriquettes*, he states that "the oldest coke-oven employed for recovering tar

products is that of Carl Knab, of St. Denis, near Paris." For example, I have only to refer you to Fischer's *Jahresbericht*, 1883, p. 1213. However, Lunge in his work on the Distillation of Coal-tar, p. 14, refers to Stauf and his coke ovens or muffle furnaces of 1768, but in doing so he quotes from p. 22, of Dr. Gurlt's work already mentioned, which brief reference would almost seem to have escaped other authorities as Hüssener and Fischer.

Knab originally (about 1856) worked only with bottom flues beneath the bed of his coke oven, according to Hüssener; and at the coke ovens of Lebrum at Commentry, Carvès, under whose management, at St. Etienne, 88 Knab ovens were erected, first introduced the heating of the ovens with side flues in addition to that with the bottom flues. After the improvements of Carvès, there were gradually introduced at Besseges 53 coke ovens in the years 1866 to 1873, and in the year 1879 at Terre-Noire, near St. Etienne, 100 ovens. However, according to Dr. F. Fischer (Fischer's *Jahresbericht*, 1883, p. 1214) there were already, in the year 1854, fifty coke ovens known as Pauwels-Dubochet ovens at work near Saarbrücken, which would appear to make these ovens of somewhat earlier date than those of Knab and Carvès. On this point Dr. Gurlt informs me (Oct. 10, 1884) that the Pauwels-Dubochet coke ovens were certainly in use in 1854 at the coke works of De Wendel, near Saarbrücken, but no tar was ever produced from them, all the gases and products of distillation being burnt around the coking chamber to heat it. The walls of the coking chamber were inclined at an angle of 45°, and the oven had an auxiliary fireplace near its lower opening, through which opening the coked charge could glide down into a cooling chamber. This oven must hence have borne considerable resemblance to the more modern Appolt's oven.

Later on, it would appear that the Pauwels-Dubochet coke ovens were improved, possibly as the result of Carvès' improvement of the Knab ovens, for the alterations effected in the introduction of side flues as well as bottom flues, and the cutting off these flues from connection with the interior coking-space, so that by-products and gas could be recovered, were identical in both cases.

In the beginning of the year 1870, when coke rose in price immensely, the Chief Director of the Paris Gas Company, W. Oechelhäuser, caused the introduction at the stations Ivry and La Villette of two sets of Pauwels-Dubochet ovens, for the purpose of producing from caking-coals metallurgical coke and illuminating gas for the city of Paris, one-half of which was supplied by the latter. It was said to have less illuminating power than good retort gas, for which reason it was mixed in the gasometers with retort gas. The coke was said to be the best imaginable, and commanded a high price for use on locomotives and for metallurgical and other purposes. The dimensions of the fireplace of one of these ovens as given in Gurlt's work (p. 30) are—length, 7·20 meters, breadth, 2 meters, and height of vault, 1 meter. Dr. Fischer, however, gives for Carvès ovens, as erected at Besseges *Jahresb.*, 1883, 1214, the following particulars:—"These ovens were (according to many measurements) only built 0·6 meter wide;" and Gurlt adds as to the Pauwels-Dubochet oven, "The working of this oven is generally the same as the Knab oven (Carvès oven), only the coking of the charge of six tons takes place at a very much lower temperature and lasts for three days, whereby one ton of coal yields 7,500 cubic feet of gas." But these differences, however slight they may appear, are fully sufficient to make a complete difference in the tars obtained; and Gurlt further on remarks, as he might be anticipated to do, "The tar is much lighter (*i.e.*,

has a much lower specific gravity) than gas-retort tar of a specific gravity of 1·2; also it contains less naphthalene and carbolic acid." I feel no doubt myself that the narrower-built Carvès oven would have yielded a better illuminating gas than did the Pauwels-Dubochet oven, but the coke would have been harder.

The earlier Carvès oven was furnished with a fireplace and grate, the uncondensable gases being brought round to the fireplace by pipes, and burnt there. Otherwise the flue arrangement was much the same as now. The recent Carvès oven is fired exclusively by the gases escaping condensation, these entering the lower flue at the place where the hearth or fire used to be, air being forced in through an annular pipe. In the most recently developed *Simon-Carvès Oven* or *Recuperator Oven*, the air introduced receives a previous heating to some 500° to 600° C. (900° to 1,000° F.) by the flues being brought in contact with the hot flues conveying away the spent fire-gases from the ovens.

I consider from all the evidence obtained, that to M. Carvès belongs the full credit of being the first to construct and work a coke oven capable of yielding good metallurgical coke, coal-gas, and valuable coal-tar, which with astonishingly little modification, continues to the present time the simplest and best oven for the purposes named, according to my opinion.

With regard to the coal-tar furnished by the Simon-Carvès coke ovens, I have a quantity of distillates taken at different stages of the tar-distilling process, under examination, and since I hope to place the results of this investigation before this Section some time in January, I will not anticipate that communication, but merely be content to mention that the tar of the Simon-Carvès process containing as it does, almost three times the quantity of anthracene contained in ordinary gas-retort tar, will form a valuable source for the alizarin colours, the formation and fixing of which on woollen fibres has been so ably explained in the paper of my friend Mr. Hummel. Thus, it has at length become possible that in order to do full justice to all possibilities of profitable working and business, the colliery proprietor and metallurgist may yet be found linked to and connected with the colour manufacturer and dyer. I will conclude by giving one practical proof of this. From a gallon of ordinary solvent naphtha derived from the tar from the Simon-Carvès coke ovens, which naphtha I have been for some time quantitatively fractionating, I have obtained a very considerable proportion of pure commercial xylenes boiling between 136° and 142°. A determination as to the presence or absence of the commercially valuable metaxylene in the mixture of isomers, has shown me that this xylene is as rich in metaxylene as the best coal-tar xylenes. The truth of this statement, and the value of the process devised by Mr. Levinstein for estimating the proportion of metaxylene in samples of xylenes, was borne testimony to by the fact that on sending a specimen of my product to Mr. Levinstein, and asking his manager, Dr. Rosicki, to report as to the colour-producing value of the xylene sent, he replied by returning me the following products, which I have pleasure in exhibiting: (1) Sample of nitroxylene, (2) sample of xylidine, and (3) sample of xylidine scarlet with piece of woollen cloth dyed with the scarlet, and a note in which he gives me data proving that the quantitative results I obtained as regards ratio to amount of colour he obtained are indeed a valuable guide for a dye manufacturer buying xylenes.

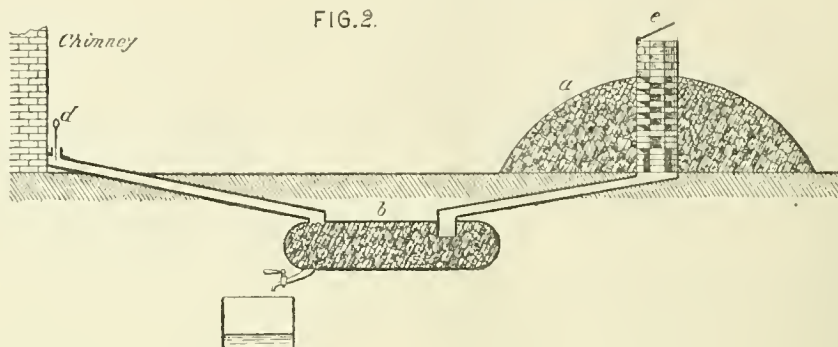
#### ADDENDUM.

To give you an idea of some of the earlier efforts



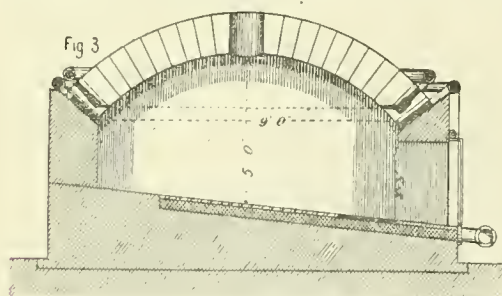
put forth in England and Scotland to coke coal for metallurgical coke so as to gain ammonia and tar at the same time, I would draw attention to figs. 2, 3, and 4; fig. 2 illustrates an arrangement contrived by Edward Jones between 1850 and 1860. I believe, for burning in pile and recovering tar and ammonia water.

densing these vapours to obtain tar and ammonia water, seems to have occurred to Aitken in 1873. The following words are recorded in his paper read before the North of England Institute of Mining and Mechanical Engineers in 1874. "Few coals have more tar in them than is required to make good coke, but



*a* is the pile, the central chimney of which has a damper *e* closed during carbonization so as only just to admit sufficient air, *b* is an old boiler packed with

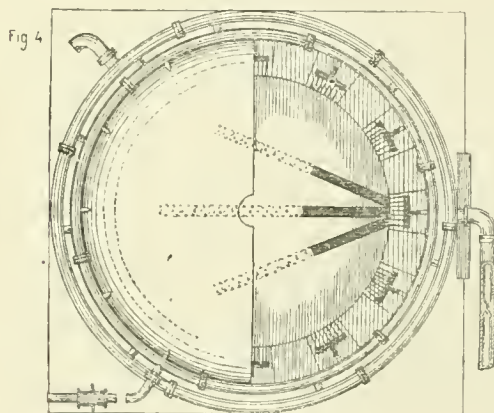
where such a coal is made into coke these tars may be drawn off by a pipe or pipes at the bottom of the oven, the gas exit being partially closed, and thereby creating pressure sufficient to force the gases through the pipes. Several pailfuls of tar and ammoniacal water have, in some cases, been taken from a single oven, but the quality of the coke has always suffered." In his patent No. 57, January 5, 1874, Aitken claims, besides the forcing of air into coke ovens, the drawing of it by means of an exhauster; and he further adds, "when there is more tarry matter than is necessary for 'gendering,' a portion of the same may be drawn off at the bottom of the ovens by means of pipes, and thereafter condensed, and thereby a portion of the tar and tar-water is secured." The close resemblance of Jameson's later patent to this is tolerably plain.



coals and acting as a condenser, *c* is a well to receive products run off from *b*; *d* is the chimney with damper to flue from *b*, and thus acting as an ex-

Figs. 3 and 4 refer to a more perfect process described in 1879, where Aitken writes, first contradicting his former statement quoted above, about the injury to the coke as a result of drawing off tar or coal oils and ammonia water, and now advocating the latter, and especially for some kinds of Scotch coals, and secondly, describing his new oven and process, as follows:—"In order to carry on the coking in the blast oven, while at the same time taking off the oils and ammoniacal water, three flues are made in the bottom of the oven, and these are covered with tiles having perforations about  $\frac{3}{4}$  in. diameter at top, and 1 in. diameter on the under side. These three flues meet in one near the door, and into this flue the main pipe is inserted, which is connected with the condensers. It is necessary that a mechanical exhauster or steam jet should be in connection with these in order to assist in drawing the gases from the ovens."

Although this paper is professedly to deal with the earlier records of coke ovens, yet I may be excused perhaps if I add a table showing a classification of these ovens from the earlier dates to the present. A brief study of this table, with references to the illustrations given already in this Journal, will show the course of development of more elaborate and complete forms from the simplest root forms. This table I arranged for a paper read before the Iron and Steel Institute in September of this year, and I believe it is the first attempt that has been made to classify and tabulate the various forms of apparatus employed to coke coal for metallurgical purposes.



hauster. Figs. 3 and 4 represent in vertical section and plan the adapted Beehive of Henry Aitken, of Falkirk. The idea of drawing off tar and ammonia vapours from the bottoms of beehive ovens, and con-

## TABULATED ARRANGEMENT OF THE PRINCIPAL COKE OVENS.

Bye-Products not Recovered.		Bye-Products Recovered.			Remarks on E.
A.	B.	C.	D.	E.	
		Modified Recovery of Products.	Recovery or Non-recovery of Products at Will.	Full Recovery of Products, or Uninterrupted Close Distillation.	
AIR ADMITTED, and so partial combustion in the coking space itself.	I. NO AIR ADMITTED into the coking space, but into the side flues, so as to mix with the gaseous products passing through openings in the oven walls into side flues. Thus complete combustion of all the products, and so the ovens are heated.	I. AIR ADMITTED into the coking space, and so a partial combustion; moreover, the character of the bye-products recovered is modified by this admission of air. Gases not saved or utilised.	I. NO AIR ADMITTED into the coking space, but at certain stages of the process the volatile products can pass through spaces in the walls of the ovens into side flues, and mixing there with air, be burnt to heat the oven, or at will this through-passage can be cut off, and the volatile products can be drawn off by an exhaustor and condensed, the pure gas being returned into flues and burnt with air. II. In addition to I., in some cases:— AIR ADMITTED INTO COKING SPACES towards the close of process, forming a SECOND OR FINISHING STAGE: the half-burnt gases furnished being burnt in the sides or other flues. III. Or, in addition to II., the air admitted to burn the half-burnt gas MAY RECEIVE A PREVIOUS HEATING.	NO AIR ADMITTED into the coking space, and in all cases the volatile products are drawn off by exhaustor, subjected to cooling and condensation for the sake of tar and ammonia, and the gases are— I. Returned to the ovens and burned in the flues with— (a) Air admitted cold, or (b) Air previously heated ("recuperated.") II. Or gases returned and burnt, in conjunction with— (a) Small fireplace and solid fuel; (b) Or, fire of gas-producers. III. Or, gases not returned to ovens at all, but used as illuminating gas or otherwise, the ovens themselves being heated by— (a) Solid fuel. (b) Fire from gas-producers.	
Thus some coke is burnt.	Thus coke is not burnt.	Thus some coke is burnt and also some ammonia.			
	II. HEATED AIR ADMITTED INTO SIDE OR BOTTOM FLUES.	II. In addition to I., gases saved after condensation, but not utilised in the coke-ovens themselves. III. In addition to I., gases, after condensation, return for combustion, and are burnt in flues around the ovens or otherwise, to heat them.			
Meiler or mound . . . (1) Beehive oven . . . (2) Lürmann's oven . . . (3)	I. Jones' coke-oven . . . (1) I. Appolt oven (2) I. Coppee oven (3) I. Sachse oven (4) I. Hiltawski oven . . . (5) I. Franzen oven . . . (6) II. Müller oven (7) II. R. Wintzek's oven . . . (8)	I. Jones adopted Meiler . . . (1) II. and III. Jameson's adapted beehive ovens . . . (2) III. Aitken's adapted beehive oven . . . (2) III. Lürmann's oven . . . (3) (Ger. Pat. Suppl. 17,203, 1881.) II. Hutchinson's adapted beehive oven . . . (4)  Under head Comes also— I. Recovery of tar and ammonia from blast furnaces fed with coal . . . (4) Alexander & McCosh. See specifications and drawings in Eng. Pats., 4117 for 1879, 1433 for 1880, 3785 for 1881. III. Recovery of tar and ammonia from gas-producers (5) W. S. Sutherland.	III. Klönne's coke-oven . . . (1) (Kind of adapted Beehive.)  I. Otto's oven (adapted Coppee) . . . (2) III. Lürmann's oven (3) (Ger. Pat. 20211, 1882.) II. R. de Soldenhoff's oven . . . (4) (Ger. Pat. 25,824, 1883.)	Stauf's coke-oven about (about 1764) . . . (1a) Knab's coke-oven (1855) . . . (1b) III. a. Pauwels-Dubochet oven, constructed similarly to Carvès oven (1854) . . . (2) II. a. Carvès oven (Carvès and Haupt) (modified Knab) . . . (3) I. b. Simon-Carvès oven (Recuperator) . . . (4) II. a. Pernolet's adapted Beehive . . . (5) II. a. Pernolet's earlier coke oven . . . (6) II. a. Pernolet's coke oven . . . (7) I. a. or b. Adapted Coppee oven . . . (8) II. a. Otto's coke oven . . . (10) II. b. Herberz and Otto's coke ovens . . . (11) I. b. Hussener's coke oven . . . (12) I. b. Ruppert's coke oven . . . (13) I. b. II. b. or III. c. Herberz's oven . . . (14) I. a. and b. Semet and Solvay's coke oven (15) I. a. Seibel's coke oven . . . (16) I. b. Regenerative coke-ovens of the Coal and Coke Works, Gottesberg . . . (17) Maderspach and Sierch's coke-oven (1883) adapted Appolt with alternative regenerative gas furnace heating (18)	(1a.) See description, page 18. (1.) At first not adapted to recovery of products (all volatile matter being returned to fire the ovens) Later modified for this purpose by Carvès and Haupt. (5.) See "Musparratt's Dict." iv. 959 and 960. (8.) See "Fischer's Jahresh," 1883, 1216 Modified Carvès oven. Gas in side flues, and fired below by band-fire in fireplace. (10 and 11). Still more recent forms than in D. (12). Modified Simon-Carvès oven. Gas admitted in fireplace and side-flues with air heated to 300°. (13). Ruppert has cold air and regulative arrangements. (15). Slightly modified Simon-Carvès oven. (16). Also slightly modified Simon-Carvès oven, and very similar to Semet & Solvay's oven. (17). Has an ingenious alternative regenerative heating arrangement for gas and air, or in some cases of air alone.



Tar Valuable for Dyes and Colours.	Tar not Valuable for Dyes and Colours	Tar probably Valuable, but Definite Data not to hand.	Doubtful as regards Tar.
..	*C (2)	..	C (1)
..	*C (2')	D (3)	C (3)
..	*C (5)	D (4)	C (4)
..	*E (2)	D (2)	D (1)
*E (3)	*E (5)	..	
*E (4)	*E (7)	E (8)	
E (8)		E (9)	
E (12)	..	E (10)	
E (13)	..	E (13)	
E (15)	..	E (18)	
E (16)	..		
E (17)	..		

## DISCUSSION.

MR. H. GRIMSHAW: I should like to ask Mr. Watson Smith if nothing has been done in England with regard to the study of coke ovens, whilst what Mr. Smith has described has been carried out in Germany. Probably practical experiments of some description have been carried on in this country, but from that want of scientific method in our work which has been an unfortunate feature of so much English experimenting, the results of all this have not been placed on record. This same absence of scientific method in English work has been commented on in the course of our Chairman's admirable address, and I think the moral that we should gather is that the great utility of a society like our own is to impress upon the chemists of this country the importance of that union of science with practical experience, which is necessary in these times to keep us to the front in our industries.

DR. HEWITT: It is evident from Mr. Watson Smith's paper that the first attempt at the utilization of waste products from coke ovens arose from a desire to deal with what had proved a disagreeable nuisance, by polluting the atmosphere, and this is by no means an isolated instance of great results following from attempts to utilise the waste products of various manufactures. Sir Henry Roscoe had well suggested that there remained much more to be done in this direction. To anyone who takes an interest in this subject of coke ovens he might mention that the Society's Journal contained a complete repertorium of facts and details extending from their earliest use to the present time, and that information would be found to relate to English, German, and French coke ovens.

\* All the above are tabulated from personal experience, or from reliable information.

E<sub>1</sub>, E<sub>12</sub>, E<sub>13</sub>, E<sub>15</sub>, E<sub>16</sub>, and E<sub>17</sub> are judged to yield valuable tar from the known construction of the ovens and nature of firing processes, and from the fact that they are all modified Carvès ovens.

The other forms tabulated are merely judged of from plans showing construction and details as regards firing (temperature).

References where plans can be seen, short accounts read, or references to patents obtained, etc.:—

This Journal, vol. ii. 322.

.. vol. iii. 101.

.. vol. iii. October 1881, 510 to 512.

*Jahresbericht über die Leistungen d. Chem. Tech. für das Jahr 1883* (Dr. F. Fischer), xxix. p. 1213.

*Muspratt's Dictionary*, iv. 953 to 966.

*Gurtt, Die Bereitung d. Steinkohlenbriquettes*, 1880, 28.

*Dr. Percy on Fuel*, p. 423 et seq.

MR. WATSON SMITH: In reply to Mr. Grimshaw's question, whilst echoing in the main Dr. Hewitt's closing words about our Journal, I would briefly remark that in the coking industry the French have proved their superiority in earlier times in the direction of the original devising of apparatus for coking coal over both Germans and English, as the names of Carvès, Hauptart, Pernolet, Coppée, Pauwels, and Dubochet, bear testimony. But the name which stands out brightest is that of the man who first practically recognised that certain high and special thermal conditions concurrent with the use of a closed carbonizing space, were necessary to give at once good coke, ammonia, and tar rich in aromatic substances, and who persevered to the end, it is that of Carvès. Otherwise the English coke ovens possess great merits, as also do the German ovens, looking away from the tar and ammonia as regards quality and quantity. However, I will, if I may be permitted, make an addendum to my paper, so as to answer Mr. Grimshaw's question by one or two illustrative cases.

## Newcastle Section.

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Notices of papers and communications for the meetings to be sent to the Local Secretary.

MEETINGS, SESSION 1885, Jan. 8th, Feb. 5th,  
March 5th.

Three short papers by Professor Lunge, of Zürich, will be read:—1. Reducing action of coke on nitric acid dissolved in sulphuric acid. 2. Solubility of calcium sulphate in solutions of sodium and calcium chloride and in dilute hydrochloric acid. 3. Action of chlorates on metallic evaporating vessels.

General Meeting held in the Lecture Theatre of the College of Science December 4, 1884.

MR. J. C. STEVENSON, M.P., IN THE CHAIR.

## DISCUSSION ON THE CHAIRMAN'S ADDRESS READ AT THE OCTOBER MEETING.

THE CHAIRMAN: The question of the comparative advantages of machinery and hand labour is one of ever widening range of interest, and my object in making it the subject of my address was to elicit if possible the opinions of members who look at the question from very various points of view. I submit the following remarks as a contribution to the same subject, and I invite discussion, and trust that the circumstance of its being unusual to remark on a presidential address will not cause members present to have any hesitation in giving it their fullest criticism. Many attempts have been made to save hand labour in the firing of steam boilers by the adoption of mechanical appliances. The inducement to adopt such contrivances generally is that by the uniform supply both of air and fuel the conditions of

complete combustion can be maintained with consequent economy of fuel and prevention of smoke, combined with the use of small and inferior coal. I believe that the prevention of smoke is the only certain result of all these attempts, and that has been attained only when the fire has been kept within moderate bounds—heavy firing producing smoke just as in an ordinary fire. I gave some years ago a fair trial to Jukes' furnaces, but found that the consumption of fuel for the same boilers was much increased. I was told afterwards that I should have used short boilers and brought the heat back through tubes within the boiler. But this was only another way of stating that for ordinary bottom-heated round boilers Jukes' furnace was not suitable; while in the nature of the case it cannot be used in a tubular fire place within the boiler. I tried Vickers' patent where the bars are moved backwards and forwards, and are prevented from getting too hot by having webs attached to them which are immersed in troughs of water. The troughs, however, filled up by deposit from the evaporation of the water, and the system was otherwise a failure. In all such cases the power to actuate the machinery is an element of cost as well as a cause of occasional stoppage and repair. In chemical works where the boilers are wanted day and night the work is more severe than in factory boilers where the work is suspended during the night, and the apparatus has time to cool, and can be examined and slight repairs effected. I have found after 20 years' experience that the best boiler is Galloway's, with two fireplaces joining into one large flue crossed by conical tubes. These boilers make no smoke if the two fires are not coaled at the same time, only moderately careful stoking being required, and any waste of heat can be due only to an unnecessary amount of draught by the chimney. If stoking by machinery were practicable it would be in steamships that we should expect to see it first adopted, for in the stoke-holes of steamers the labour of firing is necessarily much more severe than in boilers on land.

Mr. STUART: Although Mr. Stevenson has not committed himself to figures in his admirable address, yet he has gone very fully into the comparative advantages of machinery, as a substitute for hand labour in alkali works, comparing revolvers, mechanical carbonators and mechanical decomposers, with their respective hand furnaces; and contrary to the usual custom, he has invited us to discuss the question to-night. This subject is, or ought to be, a purely financial one, and should be argued on that basis alone; but notwithstanding that I have had long experience in this question, and ample opportunities of forming matured opinions, I regret that I am unable to state the relative merits of these furnaces exactly in figures. The advantages and disadvantages on both sides are of two kinds. First of all we have the direct advantages, which can be easily stated in money, then we have the indirect advantages which cannot be so represented. To begin with, let us follow the Chairman's address, and discuss the relative advantages and disadvantages of revolvers, as against hand furnaces, and then we shall recognise the obvious difficulty of stating exactly what the saving is per ton of sulphate balled. The disadvantages of revolvers are, that per given quantity of work done, they cost when every item is carefully taken into account, considerably more than hand furnaces, there is no saving of coal or limestone, and so far as Tyne practice is concerned, the colour of the finished soda ash, is not so good as that produced from hand-made balls. Then the cost for repairs and stores is higher; but on the other hand, it only costs in round numbers for labour 1s. per ton on sulphate, as against 3s. per ton in hand furnaces,

showing a saving of 2s. per ton. In addition to this, we have the indirect advantages, to which it is very difficult to assess an exact money value, but which have an important financial bearing on the cost of manufacture, and consist of better decomposition of the sulphate, stronger ash and better yield. Then you dispense with a large number of troublesome men, bring the operation of balling under easy control strikes to a large extent are avoided, while large quantities of materials are manipulated with ease and comfort to all concerned, and regular and certain results obtained. I quite agree with Mr. Stevenson that while revolvers have proved a success, and that none of us in large works at least, would like to go back to hand furnaces, yet the direct saving is not much, and certainly not sufficient as a rule to justify their erection in small works. We have from time to time had occasion to make comparative costs of revolvers, mechanical carbonators and decomposers, against hand furnaces, and I have done so again for the purpose of this discussion, but based on the present prices of raw materials and labour, and after going into the matter very carefully, I can only make the direct saving in favour of revolvers to be 9d. per ton on sulphate, or say 1s. per ton on 48 per cent. ash; but when this is combined with the indirect advantages already enumerated, I think that the revolving ball furnace has proved a success, warranting its general adoption by the trade. Mr. Stevenson next discusses the merits of the mechanical carbonator, and I am at one with him in regarding this machine as not nearly so scientific as the hand furnace. Like all other mechanical furnaces, it costs more for plant per ton of ash carbonated than hand furnaces, and it is not nearly so efficient in oxidising the sulphides. It also rolls up a large proportion of the ash into round fluxed pieces, which dissolve very slowly when used for soda making, and which are liable to pass through the dissolver, settlers and mud washers into the drains if not carefully treated. In order to completely dissolve these fluxed pieces, we find it necessary to boil the mud washers 14 times, 12 hours each time, and if this is not done a serious loss in yield results. There is no saving in fuel, as the Chairman points out, and the repairs amount to 8½d. per ton as an average of four years' working. On the other hand the ash being better carbonated, and containing less caustic soda, stands a higher temperature in finishing, and the colour is decidedly better. Indeed if sufficient time be allowed the ash can be finished up to the colour almost of white alkali. Then another advantage of the mechanical carbonator consists in the fact that the difficulties in making good-coloured soda are at once overcome, for the traces of oxide of iron which so effectually discolour soda, seem to be made so dense by the high temperature to which they are exposed that they settle out completely, leaving a clear, well-settled liquor, which gives perfectly white soda. Then finally, as Mr. Stevenson points out, the ash is more dense, and there is a consequent saving in casks. Here again, as in the case of the revolvers, we have the same difficulty in stating precisely the saving per ton derived from the mechanical carbonator over hand furnaces, as we have direct and indirect advantages and disadvantages associated with both. The cost for labour is 1s. per ton as against 3s. in hand furnaces, leaving a balance of 2s. in its favour; but against this there is a higher cost for interest on plant, fuel and repairs, and when these are all carefully considered we make the present saving about 1s. per ton on ash, which added to the 1s. saved in the revolvers makes a total direct saving of 2s. per ton of 48 per cent. ash, as a result of replacement of hand labour by revolvers and mechanical carbonators.



Standing by itself, I don't think any manufacturer would be induced to alter his plant for this small saving; but combined with the indirect advantages of fewer men, comparative immunity from strikes, the ease with which large quantities of materials are manipulated, better decomposition of sulphate, better strength, yield and colour of ash, I am of opinion that all these with the 2s. already shown, would represent at present a total saving of 5s. per ton of ash, which I think perfectly justifies the expenditure incurred by the trade in the past, for the substitution of hand labour by means of revolvers and mechanical carbonators. Mr. Stevenson next considers the bold attempts to solve the problem of the application of machinery to the decomposition of salt by means of the two forms of mechanical furnaces with which we are all familiar, and expresses the opinion, which I perfectly agree with, that these plans will not be generally adopted by the trade. Mr. Stevenson also remarks that the evolution of two-thirds of the acid by the low heat of the pan, and of the rest by the higher heat of the drier, will always be better than using one furnace for the whole operation, and if he were disposed to experiment on mechanical decomposition, he would not give up the pan which does so well so much of the work, but would supplement it by a mechanical roaster. This is precisely what has been done by Messrs. Black and Larkin, who have two of their furnaces successfully at work at the St. Bede Works, South Shields. To an ordinary pan is attached a mechanical close roaster worked with rotating arms, as in the earlier pattern of the Jones furnace. This furnace is cheap to erect, makes good sulphate, requiring cheap fuel in the form of small coal, and the pan roaster acids are evolved at a low temperature, and being undiluted by the products of combustion or air, very satisfactory condensation and strong hydrochloric acid are obtained, and no wash towers are required. If the mechanical decomposition of salt is insisted upon, I quite agree with the Chairman in thinking that this is the principle upon which the furnace should be erected. Revolvers and mechanical carbonators have each only one operation to perform—to ball and carbonate; but the decomposing furnace has two distinct operations, to make sulphate satisfactorily, and evolve the hydrochloric acid in that condition in which it is easily condensed into liquid acid of high specific gravity. No decomposing furnace, whether hand or mechanical, will satisfy the requirements of the present day, unless 98 per cent. of the hydrochloric acid evolved can be condensed without wash towers, and acid obtained of 30° Tw. for the importance of manufacturing bleaching powder from the minimum quantity of salt is becoming obviously greater every day. The open mechanical decomposing furnaces recently introduced, in addition to making satisfactory sulphate, and removing the low level gas, have proved a mechanical success; but financially and chemically they have not realized the anticipations of the inventors; financially, because when compared with the best form of hand furnace there is no saving, and chemically, because of defective condensation. These furnaces effect a saving in labour similar to that made by revolvers and mechanical carbonators, doing the work for one shilling per ton on sulphate, as against, say three shillings by hand; but this is counterbalanced by the interest for the excessively high cost of the furnace, high cost for fuel in the form of coke, high repairs, and royalty in addition. All these together prevent these mechanical furnaces from showing a saving when compared with the plus pressure hand furnaces, which have been so wisely and so generally adopted in Lancashire. These plus pressure furnaces are very cheap to erect, as compared with

open mechanical furnaces, per ton of sulphate made, small coal is used as fuel at, say four shillings per ton, as against coke at ten shillings, and repairs and royalty are less. As the value of hydrochloric acid has gradually increased, so I think has our knowledge of the best conditions for its condensation. With the plus pressure hand furnaces now so generally adopted in Lancashire, and which are being rapidly introduced on the Tyne, the pan and roaster gases are evolved at a low temperature, and in a highly concentrated form, by which 98 per cent. of the acid evolved is easily condensed to liquid acid of 30° Tw. cold, no wash towers being required, which is important in Lancashire, where the wash tower acid is liable to meet tank waste liquors in the drains, causing a nuisance from the evolution of sulphuretted hydrogen; and the escapes do not exceed 5 of a grain of hydrochloric acid per cubic foot, on the average. With the open roaster on the Tyne we know how easy it is to condense the concentrated gas evolved from the pan, which constitutes two-thirds of the whole; but we also know how difficult it is to condense the remaining open roaster gas, which is highly heated and highly diluted with the products of combustion and excess air. With the plus pressure hand furnace, the whole of the gas is comparatively cool, concentrated, and easily condensed; with the Tyne open hand furnace only one-third of it is in the condition of roaster gas, but with the open mechanical furnaces the whole of the gas is in the roaster condition, highly heated, and largely diluted with the products of combustion and excess air. This, to my mind, is the secret of the defective condensation from these mechanical furnaces, the acid being only about 20° Tw. cold, by which an additional loss is sustained in the still liquors, against 30° Tw., and the condenser escapes  $2\frac{1}{2}$  to 3 grains per cubic foot, against 5 with the plus pressure hand furnaces, rendering wash towers indispensable, which are wholly unnecessary with the plus pressure furnace. As the efficient condensation of hydrochloric acid to a liquid acid of high specific gravity depends upon its evolution at a low temperature, and in a concentrated form, the decomposing furnace of the future, whether hand or mechanical, must obviously be a close one; if mechanical it must be done in two operations, for it is difficult to imagine a close mechanical decomposer doing the work in one operation, and the inner bottom of which would stand the batch when in the boiling condition. The very opposite conditions exist, on the other hand, in connection with the open mechanical decomposers now in operation, from which the hydrochloric acid is evolved, highly diluted, and heated with the products of combustion and air, and therefore in the most difficult condition for obtaining satisfactory condensation and strong acid, which is so important in the manufacture of bleaching powder. I am of opinion that the results obtained from the introduction of roasters and mechanical carbonators have warranted the amount of money that has been spent upon them; but one cannot say so much for the open mechanical decomposers, which in connection with the all important question of condensation are, in my opinion, wrong in principle. I should like to follow Mr. Stevenson in discussing the other applications of machinery as a substitute for hand labour, had time permitted. I agree with him in thinking that the introduction of machinery should only be yielded to after ample demonstration of the clear preponderance of advantage, when all the details are carefully investigated; also that there is a limit to its profitable application. In the present condition of the Leblanc process, when we are all anxious to cheapen the cost of manufacture in every way, I think Mr. Stevenson has done well to raise the question as to how far the application of machinery can be made a profitable sub-

stitute for hand labour, and having so frankly expressed his own views on a subject about which it is extremely difficult to get practical men to agree, I think he deserves the best thanks of the Society.

Mr. W. J. WARNER, of the South Shields Gas Works, was introduced to the meeting by Mr. Stevenson, and said: Having been very kindly invited to give on this occasion my "Experience as to the Advantages of Machinery in Gas Works," I venture to express a hope that my remarks may be of some service to the members of this Association. But first allow me, Mr. Chairman, as a gas engineer, to acknowledge my obligations to you for your kindly reference to the recognition by your Society of gas engineers as representing an important department of chemical industry. We certainly have very much in common that is of interest and importance. As a proof of this, nearly every subject treated upon by the Chairman in his interesting address are subjects that are receiving or have received considerable attention from gas engineers: the construction and working of furnaces, the steam jet, the handling of raw material; and the travelling plates used in the Hargreaves' sulphate process were used by Samuel Clegg above 50 years ago for carbonizing coal, and constituted, I may say, the first machinery for carbonizing purposes in gas works. The condition of the problem (the substitution of machinery for hand labour), which you lay down is that there shall be a clear preponderance of advantage, considering first cost and current expenditure, of the mechanical appliances. This is the condition, but in a manufacture such as ours, I presume the word advantage would not be limited to a lessened cost of production, only to a monetary advantage. A lessened risk in the production of an article which is often used as made, with no stock, no reserve to fall back upon, and which must be supplied, is an advantage the authorities of every gas undertaking incur a grave responsibility in not availing themselves of. There are other advantages which must flow from the employment of machinery for carbonization, but I need not refer to them further—this advantage is paramount. In the course of the address reference was made to a small soda work in Lancashire, in which there is not a single steam engine except for feeding the boilers. This is the case, too, with some very small gas works, but they are the exception. In works that may be fairly so called, there are boilers, engines, tools, and mechanics. I look, then, upon the introduction of machinery as but a development; the transition may not be a satisfactory stage, but if the way has been carefully felt, success, though it may be retarded by opposing interest and prejudice, will be attained. As an instance of such a development in the purifying house of a gas works, a travelling crane, or other means of raising and shifting the covers of the purifiers, must be employed. With us, at the Shields station, this is done by five men, who also discharge and recharge the purifiers in about eight hours, lifting about five tons of material. At Jarrow, a travelling crane, driven by an endless rope and small steam engine, raises the cover, shifts it, and then hoists and carries the purifying material, about 10 tons; the work is double that of Shields, is done in about 10 hours, and four men only are employed. The extra cost of machinery over the other arrangements has not been £300, and this cost has been more than covered by the saving of labour alone. To pass on to the retort machinery, my experience here is the same. Hand labour will soon become a thing of the past. Our machinery gets more efficient year by year, not from any alteration in principles, but from a greater elasticity in its action and a better knowledge of its power. The work we do by the machinery

is raising the coal by hoists to feed the machine; the machine draws the coke from the retort, recharges it, and travels from retort to retort; with the same hoists as lift the coal, the coke, which has been drawn into buckets, is raised and carried through the side of the retort house, and deposited in the yard. The tools of the machine correspond with the position of the retorts, which are at equal distances along the house, so that when a retort is drawn, and the machine moved to bring the scoop for charging into position, the movement to draw the next retort carried also the rake into its position without further adjustment. This does not now hold good for all the retorts, as we have introduced two extra retorts this year into the middle of each bed, and hence, as there were originally six in a bed, we have increased the power above 30 per cent. Leaving the men at furnaces out of the question, which are less than in some other works, we have two men at the machine, one drawing and one charging, and one at the hoist. This is for each shift six men. Now we have 12 beds of 8 in a bed in action, or 96 retorts in all, for which we should require at least 14 men for usual stage working. The difference these will make at this rate is about £14 per week. Now as we may place the machinery belonging to this section of the house, we have only a half of the house at present laid out, at about £2,000; and as at present prices of our stock, we pay about 5 per cent. for our money, the interest on the amount in rental is easily met, with a very large margin for contingencies and allowances for repairs and maintenance. The whole of the machinery is the same as that with which we started about four years since. This season we have scarcely had an adjustment since we started. I beg to thank you, Mr. Chairman and gentlemen, for the privilege of addressing this learned society.

The discussion on Prof. Divers' Paper, read in November, was postponed until the next meeting.

Mr. Wm. Martyn read a paper on "Chemical and Metallurgical Industries in the United States," the publication of which is deferred.

ERRATUM.—In the January number of this Journal, page 19 line 35 from top, for "discussion" read discretion.

## Birmingham and Midland Section.

Chairman: Alexander M. Chance.

Vice-Chairman: W. A. Tilden.

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A. BOSTOCK HILL, 11, Temple Street, Birmingham.

Meeting held December 3rd.

MR. A. M. CHANCE IN THE CHAIR.

THE CHAIRMAN opened the proceedings by stating that he had been asked to bring to the notice of members of his Section an appeal issued in London





has been read before the American Association of Mining Engineers by Mr. F. P. Dewey. (Reprinted in *Iron* for 1883, vol. xxii, p. 376.) But as the facts there described relate only to American coals and coke, and as the experimental method used by the author differs in some respects from my own, I think it may be worth while to lay the results of my enquiries, though incomplete, before the Society.

The process adopted by Mr. Dewey for determining the actual density of the substance of coke consists in weighing small selected pieces of 20 to 40grms. in weight, first in the dry state, then in water, after extracting the air as completely as possible by the aid of the air pump and by boiling. A further weighing of the wet and saturated coke after removal from the water gives the weight of water absorbed, and consequently a measure of the porosity.

It appears to me that whilst this process presents some advantages, and certainly appears to have been carried out by Mr. Dewey with considerable care, it is liable to several sources of error. In the first place it might easily happen that the piece selected would not represent the average quality of the mass from which it was taken, and secondly the difficulty of completely extracting the air from a lump of 30 to 60 or more cubic centimetres in volume is so great that I doubt if it could ever be completely accomplished in this way.

My object has been two-fold. I wanted first to know the volume weight of coke as it occurs in bulk, and secondly the absolute density of the substance itself irrespective of porosity.

The accompanying tables give the weight of the bushel stricken (not heaped up) of the several varieties of coke mentioned. The weight was obtained in each case as the average of three distinct operations, the measure being emptied out and re-filled after weighing. To get as fair an average as possible the coke was weighed in three sizes: (1) large as it came from the retort or oven; (2) broken so as to pass through a two or three-inch ring or riddle; and (3) smaller lumps 1in. in diameter without dust.

WEIGHT PER BUSHEL GAS COKE.

	Name of Coal.	Size of Coke.	Weights.			Mean.
1	Wharfedale.	Large.	29	29	28	28'6lb.
	"	2-inch.	30	31	31	30'6lb.
	"	1-inch.	33	35	34	34lb.
2	Aldwarke.	Large.	28	27	27	27'3lb.
	"	2-inch.	29	28	29	28'6lb.
	"	1-inch.	30	30	31	30'3lb.
3	Eckington.	Large.	30	31	32	31lb.
	"	2-inch.	33	33	32	32'6lb.
	"	1-inch.	34	33	33	33'3lb.
4	Mixture.	Large.	32	34	33	33lb.
	"	2-inch.	33	35	35	34'3lb.
	"	1-inch.	39	40	39	39'3lb.
5	Mixture.	Large.	32	32	29	31lb.
	"	2-inch.	29	32	30	30'3lb.
	"	1-inch.	32	31	33	33lb.

NOTE.—Nos. 1, 2, 3—No water used to extinguish. No. 4—Had been exposed to the weather. No. 5—15 hours in the retorts. No water used.

WEIGHT PER BUSHEL HARD COKE.

	Name of Coal.	Size of Coal.	Weight.			Mean.
1	Durham	Large.	31	32	31	31'3lb.
	(Ord. Beehive).	2-inch.	35	35	35	35lb.
		1-inch.	34	35	34	34'3lb.
2	Durham	Large.	32	30	31	32lb.
	(Weathered).	3-inch.	36	36	36	36lb.
		1-inch.	37	36	38	37lb.
3	Durham	Large.	27	29	32	29'3lb.
	(Jameson).	3-inch.	32'7	31'2	32	32lb.
		1-inch.	31'25	—	—	31'25lb.
4	Durham	Large.	39	45'5	39	41'1lb.
	(Simon-Carvés).	3-inch.	31'5	35'5	33'5	34'5lb.
		1-inch.	45'5	40'5	41	42'3lb.
		Repeated.	38'5	39'7	39'5	39'2lb.
5	Glamorgan.	Large.	39	38	40	39lb.
		2-inch.	41'5	41	41	41'2lb.
		1-inch.	44	44'5	44'5	44'3lb.

NOTE.—No. 3—Made by Messrs. Bell Bros. at the South Brancepeth Colliery, Durham, from a mixture of 3 parts Brochwell with 2 parts Busty Seam in an ordinary lift, beehive, exhausted according to Jameson's process. No. 4—Made by Messrs. Pease at their West Collieries, Crook, Durham.

In order to determine the absolute density, a mass of considerable size, or usually several lumps, selected from a heap, were taken and powdered to coarse powder. This was well mixed together and a portion of it removed and ground in a mortar to fine powder. It was then dried in a steam oven. After being placed with distilled water in the specific gravity bottle it was alternately heated and placed under the exhausted receiver of the air pump till all the air had been removed. Obviously air is expelled from the powder much more readily than from the same material in mass.

Although coke in lump floats in water the fine powder sinks instantly, but that it still contains air is shown by a comparison of the density determined without the use of the air pump with the result obtained after thorough exhaustion. In one experiment the density observed was 1'82 instead of 1'95, and in another experiment 1'76 instead of 1'80.

#### DENSITY OF COKE BY SPECIFIC GRAVITY BOTTLE TEMPERATURE, 16° to 17° C.

	Expt. 1.	Expt. 2.	Mean.
Gas Carbon .....	2'028	2'010	2'019
Gas Coke .....	1'780	1'790	1'785
Welsh Foundry Coke....	1'860	1'870	1'865
Durham .....	1'920	1'900	1'910
Durham (Jameson) .....	1'957	1'961	1'959
Durham (Simon-Carvés)	1'800	1'810	1'805

#### DENSITY FROM THE BUSHEL WEIGHT.

Gas Coke (average) .....	3'975
Hard Coke (average).....	4'400

NOTE.—The density of coke in the measure kind not mentioned, is given in Box's "Tables of Materials" as 353. The density of gas carbon is given by Mené as 1'885 (Smithsonian Collections, vol. xii, p. 27).

An interesting fact may be observed with reference to the two cokes produced by the processes of Messrs. Jameson and Simon-Carvés respectively, of which we have heard so much of late. The weight per bushel of the French coke is greater than that of the Jameson coke, but its density by the bottle is less. I thought at first that this must be a mistake, but on carefully repeating the specific gravity determination I came to the conclusion that it certainly was so in



the case of the samples in my possession. The French coke, as is well known, presents an appearance very different from that of the Jameson coke, or of coke made in an ordinary bee-hive oven, and its want of lustre may really be due to its containing a quantity of soft and sooty carbon. Whether high density is always associated with great strength and power to resist crushing, we do not yet know, and further information is desirable as to the behaviour of the new coke in the blast furnace.

I hope as time goes on to add to the facts collected in this paper. In the meantime I desire to express my best thanks to the gentlemen who have very obligingly assisted in these experiments. I am indebted for help to Mr. A. Dougall, of the Kidderminster Gas Works, to Mr. R. Dixon, of Messrs. Pease's West Collieries, at Crook, and to Mr. C. Ernest Bell, of Messrs. Bell Bros., and to Mr. Hugh Nettlefold, also to Mr. Henry Dewes, of the Mason College Laboratory for determinations of specific gravity.

#### DISCUSSION.

MR. W. W. STAVELY: I am much interested in this question of the specific gravity of coke, and I had hoped that Dr. Tilden would have given us the specific gravity of coke produced from coal tar pitch. In the tar distilleries a certain kind of pitch, which is too hard for the purpose of making patent fuel, is redistilled in specially constructed fire brick ovens, and as a result there is obtained as distillate a dense oil used for rough lubricating purposes, and left in the retorts is a very hard dense coke. An analysis of this coke which I published some years ago showed it to contain about 97½% of carbon and but one tenth of a per cent. of sulphur. Some 50 to 60 tons per week of this coke are produced in this district and used in the iron works. On account of its density and hardness it will bear a much greater load in the furnaces than ordinary Durham coke. So intense is the heating power of this coke that for ordinary purposes, it requires to be mixed with gas coke.

MR. ENOCH EVANS: Permit me to direct attention to a method for determining the specific gravity of coke, by Dr. W. Thörner, which appears in *Engineering*, November 28th, 1884, p. 495. The author used several fluids, including alcohol and benzol; he found alcohol quite suitable for ordinary purposes, and cheaper, and more agreeable to work with than benzol.

## Glasgow and West of Scotland Section.

Chairman: Ed. C. C. Stanford.

Vice-chairman: J. Ferguson.

#### Committee:

George Beilby.  
A. Crum Brown.  
T. Carnelley.  
J. Christie.  
John Clark.  
W. Dittmar.  
Geo. M'Roberts.  
Jas. Macfar.

E. J. Mills.  
James Napier, jun.  
T. L. Patterson.  
J. B. Icardman.  
Edmund Ronalds.  
F. J. Rowan.  
R. R. Tatlock.  
William Wallace.

#### Hon. Treasurer:

J. J. Coleman, 45, West Nile Street, Glasgow.

#### Local Secretary:

G. G. Henderson, Chemical Laboratory,  
University of Glasgow.

Notices of papers and communications for the Meeting to be sent to the Local Secretary.

January 6th.—(1) Mr. Beilby will exhibit and describe "A new form of Gas Thermometer." (2) Mr. J. J. Coleman will exhibit and describe "A Gas Thermometer for taking Temperatures below Freezing Point." (3) Professor Dittmar will exhibit and describe "Schaeffer and Budenberg's Tension Thermometer." (4) Mr. J. Murrie will exhibit and describe "An Improved Thermometer for taking High Temperatures."

*The opening meeting of this Section for session 1884-85, was held in the Rooms, 207, Bath Street, on Tuesday, 11th. November.*

MR. E. C. C. STANFORD, IN THE CHAIR.

### CHAIRMAN'S OPENING ADDRESS.

MR. E. C. C. STANFORD: In bidding you welcome to the opening meeting of this, the second session of the Scottish Section of the Society of Chemical Industry, I shall only detain you a few moments while I briefly refer to the past session. Our first meeting was not held until February 12th, of this year, and in my opening address on that occasion, I ventured to predict that our contributions to the Journal would not be blank pages; as a fact I find that in the four months thereafter, of 141 pages of printed contributions, those from this Section amounted to 52 pages, or over 36 per cent., which, considering the short length of our first session, is very satisfactory, and is a proof that we have redeemed our pledges.

The members of the Society of Chemical Industry now number 1,850, and we have at present, as members of this Section, 181. Of these, 96 have become members of the Society since the formation of this section. Twelve important papers were contributed to the Section, and one special meeting was devoted to discussion on "International methods of testing and sampling," in which the subject was most ably introduced by Mr. Tatlock, and the discussion is claimed to have added to the general knowledge of this fruitful source of disputes.

In the session commenced this evening, we have the following papers fixed:—

For this evening, "The application of Cooling to the recovery of certain Salts from solution." By Mr. C. J. Ellis.

December 2nd.—"On the Chrome Tanning Process." By Mr. W. J. A. Donald.

January 6th.—(1) Mr. G. Beilby will exhibit and describe "A new form of Gas Thermometer." (2) Mr. J. J. Coleman will exhibit and describe "A Gas Thermometer for taking Temperatures below Freezing Point." (3) Professor Dittmar will exhibit and describe "Schaeffer and Budenberg's Tension Thermometer." (4) Mr. J. Murrie will exhibit and describe "An Improved Thermometer for taking High Temperatures."

February 3rd.—(1) "Bromine Absorptions." By Professor Mills. (2) "On a new system of Cooling Oils for the extraction of Paraffin." By Mr. G. Beilby.

March 3rd.—"On the processes employed in Italy for the extraction of Oils, etc., from bituminous rocks in that country." By Mr. J. Murrie.

April 7th.—(1) Annual General Meeting: (2) "Notes on Destructive Distillation," by Professor Mills. (3) "Notes on the recovery of Ammonia from Blast-furnace and Producer Gases, by Mr. R. R. Tatlock.

Papers are also promised by Mr. Remmers, "On the use of Dialysis in manufacturing processes," and by Mr. Tervet, "On the fractionation of mineral oils," and I am trying to obtain a paper on the "Piccard Evaporator," which claims to distil 35 tons of water for one ton of coal expended as fuel.

At the opening of our last session we were awaiting the report of the Royal Commission on Technical Education. This has since appeared and in it the following paragraph occurs:—"The Englishman is accustomed to seek for an immediate return and has yet to learn that an extended and systematic education, up to and including the methods of original research, is now a necessary preliminary to the fullest development of industry, and it is to the gradual but sure growth of public opinion in this direction that we must look for the means of securing to this country in the future, as in the past, the highest position as an industrial nation." The truth of this proposition must be manifest to all. No one has had more experience on this Royal Commission than Sir H. Roscoe, and no one can speak with greater authority. It is therefore extremely en-

couraging to us, as chemists, to read his address to the Chemical Section of the British Association at Montreal, for he strongly defends the supremacy of British chemists in the practical applications of our science. His deliberate opinion is, that after all that has been said on this subject we are still able to hold our own against all comers.

Since we last met together, our Section has suffered a severe loss. Dr. J. J. Dobbie (our Honorary Secretary) has been appointed to a professorship in the Welsh College at Bangor. In the formation of our Section we owe much to his great zeal and ability. Perhaps no one can appreciate his labours so thoroughly or feel his loss so keenly as myself, but I am sure there will be but one feeling amongst the members of this Section; that of much regret at the loss of his valuable services, qualified only by the thought that he is transferred to an enlarged sphere of usefulness, and that our loss will be a great gain to many students and others, who will profit by association with him. We shall all watch with great interest his future career.

In appointing Mr. Henderson to fill his place, the Committee have fortunately been enabled to select a gentleman who assisted Mr. Dobbie in his work, and who will, I feel sure, tread in his eminently successful steps.

In conclusion I beg to move the following resolution:—"That the Glasgow and West of Scotland Section of the Society of Chemical Industry hereby record their deep sense of their indebtedness to Professor J. J. Dobbie for his indefatigable services as their Honorary Secretary. They beg to offer their sincere congratulations to him in his new appointment, and heartily assure him of their best wishes for his future success."

Mr. ROWAN, in seconding the resolution, said that he could bear testimony to the unfailing ability and intelligence which Professor Dobbie had displayed in the discharge of his duties as Secretary.

The motion on being put to the meeting, was unanimously adopted; and the Secretary was instructed to transmit a copy to Professor Dobbie.



## ON THE APPLICATION OF COOLING TO THE RECOVERY OF SULPHATE OF SODA FROM THE WASTE LIQUORS OF COPPER EXTRACTORS.

BY C. J. ELLIS, F.C.S.

My object in reading this paper is to bring before your notice a waste product, which is produced in very considerable quantity throughout the country, and which, I think, merits more attention than seems to have been paid to it, and some attempt to be made to recover from it the comparatively valuable and useful salt which it contains. I refer to the waste liquor, known as "iron liquor," which is produced by copper extractors, who use the wet process. I will, in the first place, lay before you a few figures to give you an idea of the quantity of this liquor which is produced, and at present run away to waste from the various copper extracting works throughout the kingdom. I have been supplied with data on this point from some of the principal works, and from those works alone it appears that certainly not less than 100,000 gallons of this liquor are daily run away. The salt, to which I refer, contained in this liquor is sulphate of soda ( $\text{Na}_2\text{SO}_4 + 10\text{H}_2\text{O}$ ), of which there is at least from 3.5lb. to 4lb. per gallon, so that from 175 tons to 200 tons of it is daily allowed to flow away in solution, being equivalent to from 77 tons to 88 tons

of salt cake, which, taken at a price of 35s. per ton, represents value per annum of from £49,000 to £56,000. Taking this into consideration, it struck me some time ago that the matter was well worth looking into, especially as at certain works it was almost imperative that this liquor should be dealt with, owing to objections being raised by sanitary authorities and others to it being drained away into watercourses or sewers just as it left the works, without at least partial purification. With this object in view I carried out a number of simple experiments on a fairly average sample of the liquor, which contained to the gallon 3.75lb. of sulphate of soda crystals, 0.99lb. of common salt, 0.74lb. protochloride of iron, and about 7lb. of water, besides traces of other bodies. For some time, at certain works, where there was a difficulty about drainage, the iron liquors had been evaporated to dryness, and the resulting mixture of salts ignited, by which treatment most of the iron was rendered insoluble, the mass was again dissolved and the sulphate of soda recovered tolerably pure by crystallization; but working in this way was found to be far from economical, and the cost of obtaining the sulphate of soda considerably more than its value, and was only resorted to as a means of getting rid of these liquors when they happened to be a nuisance. So I determined to try a totally different method, and take advantage of the property which sulphate of soda has in greater degree than almost any other salt, of having its solubility very much altered by change of temperature, and see what could be done by cooling down the liquor. I must here put before you again a few figures, as what follows mainly depends upon them. One hundred parts of water at 94° Fahr. can hold in solution 412 parts of sulphate of soda crystals ( $\text{Na}_2\text{SO}_4 + 10\text{H}_2\text{O}$ ); at 86°, 184 parts; at 79°, 110 parts; at 77°, 98 parts; at 68°, 58 parts; at 50°, 23 parts; and at freezing point only 12 parts; so that a very slight lowering of temperature in the case of a strong solution gives a very considerable yield of crystals; a solution saturated at 94° should yield almost 97% of the crystals if cooled to 32°. The waste liquor experimented on was about equivalent to a solution saturated at 65° Fahr., and on cooling it down to 40° about 2.5lb. of the salt were always obtained from a gallon of liquor; this salt was tolerably pure, and by washing it with a spray of saturated solution of sulphate of soda a salt almost free from foreign bodies was obtained. Some samples which were analysed contained about 0.2% of common salt and 0.04% of iron. The next point to be considered is how much heat requires to be abstracted from a gallon of the liquor at 65° in order to reduce its temperature to 40° and to obtain the crystals from it. We will take as our thermal unit the quantity of heat required to raise 1lb. of water through 1° Fahr. A gallon of the liquor weighs about 12.5lb. and has a specific heat of about 0.85, so that  $12.5 \times 0.85 \times 25 = 265.6$  thermal units must be abstracted in addition to that which is given out by 2.5lb. of salt while crystallizing, this may be taken at 250 thermal units, giving a total of 515.6, to obtain 2.5lb. of the salt. About ten times this quantity of heat would require to be supplied to the liquor in order to get the same amount of the salt by evaporating it to dryness. In order to arrive at an idea of the cost of abstracting heat from a solution by artificial means, I consulted Mr. Coleman, of the Bell-Coleman Refrigerating Company, and he kindly gave me certain data to go upon. He informed me that one of his refrigerators which consumed three tons of coal in 24 hours could in that time produce cold capable of abstracting 4,000,000 of the above thermal units, which is just about one tenth of the corresponding heat which the same weight of coal can supply in practice when applied to evaporation.



However, in the case under consideration it would be quite unnecessary to make use of artificial cold for the whole of the reduction of temperature from  $65^{\circ}$  to  $40^{\circ}$ , as during a considerable portion of the year, at least half this cooling could be brought about by natural means, and as the yield of crystals is proportionately much greater between  $65^{\circ}$  and  $50^{\circ}$  than between  $50^{\circ}$  and  $40^{\circ}$ , it might be found more advantageous not to attempt cooling below  $50^{\circ}$  at all. The liquor contains about 53 parts of salt to 100 parts of water, and by cooling to  $50^{\circ}$  30 of those parts should be recovered, whereas further cooling to  $40^{\circ}$  would only yield from 6 to 7 parts more of crystals. I was at first inclined to think that the question of the recovery of the salt, economically, could be solved by the use of artificial cold, produced by a mechanical refrigerator of such form as the Bell-Coleman Co. make, but on going into details of cost and working expenses, I now feel almost convinced that a similar result could be brought about in another way much more economically, and I should now like to lay before you a method I should propose for the treatment of this liquor on the large scale. Let us first take a case when the atmospheric temperature is at about  $50^{\circ}$  or lower. The liquor could be run away from the precipitating tanks in the copper works into a reservoir of suitable dimensions, where it would be allowed to remain some little time to permit of the solid impurities settling out, and also to allow the liquor to cool down to a certain extent. It could then be made to flow slowly and continuously along a shallow shoot, on the outside of which a current of cooling water ran in the opposite direction to the flow of liquor. In this way all the cooling effect of the water would be utilized, and the liquor would flow away at the end furthest from the reservoir at a temperature the same, or nearly so, as the cooling water, leaving behind it in the shoot all the sulphate of soda crystals which it was unable to hold in solution at that temperature. These could be fished out from time to time without stopping the flow of fresh quantities of liquor, and at once taken to a hydro-extractor, where they could be washed with a spray of saturated solution of sulphate of soda and dried. In this way they would be rendered almost entirely free from foreign bodies, and could then be furnace-d and converted into salt cake. This direct treatment could of course only be used when the temperature of the air and water was not much above  $50^{\circ}$ , if a fair percentage of the crystals in the liquor were to be recovered, but as this temperature is considerably below the average for a great part of the year, the liquor would require at other times to undergo treatment before entering the reservoir, so that after such treatment it would be of a strength to yield per gallon at the particular temperature as much sulphate of soda crystals as a gallon of the original liquor would yield at  $50^{\circ}$ . This could of course be brought about by a partial evaporation. Let us suppose, for instance, that the temperature was at  $59^{\circ}$ , and that the liquor was of such strength as to be capable of yielding at  $50^{\circ}$  2lb. of crystals from the gallon. From the table of solubilities at different temperatures, it is easily calculated that by evaporating away about 20% of the water from the liquor, or about 1.4lb. per gallon, a liquor would be obtained which would give per gallon the same yield at  $59^{\circ}$  as a gallon of the original liquor would give at  $50^{\circ}$ , or if the temperature were as high as  $68^{\circ}$  an evaporation of 40%, or about 2.8lb. per gallon, would again give a liquor which would yield the same result. Thus by varying the amount of evaporation according to the temperature of the air and the yield of salt required, any required result could be arrived at, the limit of course being where the evaporation was

carried to the extent of driving off all the water and leaving the dry salts. This was the case in the method of treatment which was formerly resorted to, and which, from the above, appears to be an expensive and, except in most exceptional circumstances, a useless method of procedure, for, by the method of cooling after partial evaporation, when the temperature is at  $59^{\circ}$ , only 1.4lb. of water require to be evaporated away in order to get 1.76lb. of salt (that is 2lb. less 12% for reduction in bulk of the liquor during the partial evaporation) in a fairly pure form, whereas by total evaporation, that is driving off about 7lb. of water, only 3.75lb. of a very impure salt is the result, or a little over twice the quantity of salt for five times the evaporation. Having evaporated the liquor in part, it could be run into the reservoir and be put through the same treatment as before stated. If the liquor were run from the reservoir at a temperature  $30^{\circ}$  above that of the cooling water, theoretically there would be required less than twice as much cooling water as liquor to be treated, for from one gallon of liquor weighing 12.5lb.,  $0.85 \times 12.5 \times 30 = 318.75$  thermal units would have to be abstracted in addition to 200 thermal units for the 2lb. of salt while crystallizing out, and 2 gallons of water should be capable of abstracting 600 thermal units, though, of course, in practice, rather more than the theoretical quantity of cooling water would be required. If it is required that only a given quantity of salt is to be allowed to run away in the final waste liquor for each gallon of the original liquor, and we suppose that that quantity is fixed at that which would remain in solution after cooling the original liquor to  $50^{\circ}$ ; when the temperature is at  $59^{\circ}$ , in place of having to evaporate away 1.4lb. per gallon, we should have to drive off 2.5lb. of water. Taking into consideration, however, that the liquor is a waste product of practically no value, this latter would, I think, not be such an economical way of working as the former, where a gallon of the partially evaporated liquor gave the same yield of salt as a gallon of the original liquor. The plant required for working up this waste product in this way would be somewhat as follows: Piping to run the waste liquor to an evaporating pan from the precipitating tanks in the copper works, an evaporating pan (one similar to those used in the evaporation of brine would, I think, be suitable), a reservoir, a cooling shoot, a small tank for making saturated solution of sulphate of soda, a hydro-extractor for drying and washing the crystals in, with small gas engine to work the same, a furnace for driving off the water of crystallization and converting the crystals into salt cake, and piping to run away the waste liquor when it left the cooling shoot, and to conduct the cooling water away to be used for any further purpose to which it might be applicable. I will conclude with a rough estimate of the cost of working per day of twenty-four hours, on the supposition that 20,000 gallons were to be treated in that time, and that the average temperature throughout the year is  $59^{\circ}$ . For the partial evaporation about two tons of coal would be required at 7s. per ton; say 60,000 gallons of cooling water at 4d. per thousand; four men at 4s. and four boys at 2s.; coal for the furnace, two tons at 7s.; four men at 4s. and four boys at 2s., to attend to the washing and furnacing of the crystals, &c.; gas for the gas engine, &c., 4s.; rent of ground, 5s.; management 15s.; interest and depreciation on plant at 15%, say on £2,000, taking 300 working days per annum £1; giving a total of £7. The production of salt cake for this, taken at about 2lb. of crystals per gallon, from about 17,000 gallons (after allowing for evaporation), should be over seven tons, making its cost per ton about £1.

## DISCUSSION.

Mr. E. C. C. STANFORD said: The paper which has just been read is a little difficult to follow without the figures before us, but I am certain there are several gentlemen present who can give us the benefit of their observations upon it.

Mr. M<sup>R</sup>ARTHUR: I think, Mr. Chairman, you are very correct in saying that it is difficult to follow the paper without having the figures before us. I think it is a pity that he has not made estimations with regard to other salts as well as sulphate of soda. This is certainly the one that has attracted his attention, and seems to be the most important for treatment in this way. There are few salts that are crystallizable, that are run off to such an extent as this one, and if chemistry had a conscience it ought to be troubled at the great waste. I think it is a pity that so much valuable material should be run away when it might by proper means be recovered. If mechanical refrigeration be required it must be expensive, although even in this case it would pay. I can quite well imagine sanitary or local authorities requiring solutions of this kind to be made almost perfectly free of matter in solution before being thrown into rivers and canals (canals in many cases must receive the drainage of chemical works), and in that case it would be almost imperative to use mechanical means of refrigeration, and I think that even with that it would more than pay its cost, and would yield a very good margin of profit afterwards. Statistics published recently in our own Journal show that during the last few years there has been an increased consumption of alkali, and that increase has simply been made up by the production of ammonia soda. This is a pretty fair proof that there is still a demand for salt cake, and that it would pay, so that we can consider it as being profitable and valuable. There is a method of cooling liquids that Mr. Ellis has not mentioned, and I think it also is worthy of attention. We know how rapidly heat is abstracted by simple evaporation, and I remember once, just for the sake of satisfying curiosity, putting a basin of warm water outside my window, and putting a thermometer in it, and placing beside it a registering thermometer, and in the morning I found that the water was 15° below the lowest temperature that the air had been at during the night. That was only one experiment, and I don't know what might have been the result if I had continued a series of experiments over a month. I think cooling might be done in that way, and certainly with a dry east wind a considerable amount of crystals might be got from liquors such as this. However, I think there is something more than the east wind to depend on, and so I make this only as a suggestion. I think certainly there are other salts in which refrigeration by easy natural means or by mechanical means might be profitably employed.

Professor MILLS said: I suppose that theoretically it comes to much the same thing whether you get a substance separated out by means of heat or cold. Cooling is effected by a heat engine, but universally the nature of the substance must have a very material influence, and that alone may decide as to whether we ought to apply heat or cold in any particular case. Mr. Ellis appears certainly to have made out his case, viz., economy in the application of cold in the particular case of sulphate of soda owing to the peculiar property it has of crystallising out in great abundance at low temperature. In this case, the mere application of heat raises no objection, because the sulphate is an object which you can treat as severely as you please by means of heat. This paper, however, is very suggestive in other ways, for example, in dealing with inorganic bodies

which seem to me to be specially proper substances for this treatment. Again, it is well known in the preparation of solid paraffin wax from the blue oil. In the paraffin oil manufacture cold is constantly employed for the purpose, in fact ever since the greater improvements in the paraffin oil manufacture have been made this has been systematically applied in order to extract the paraffin. It surprises me that we have not heard more of cold in this way. Why should we not purify such a substance as carbolic acid by dissolving it into some suitable naphtha, and by means of cold separate it again? By this way we might find some means of preparing carbolic acid pure with greater rapidity. Again, other polybasic acids than sulphuric acids might yield similar results. Why not try the effect of cooling solutions of benzoates and tartrates? These are some points that occur to me, and I value this paper very highly for what it suggests.

Mr. E. C. C. STANFORD in concluding the discussion said: I quite agree with Professor Mills that this is a subject which presents a very wide field. I mentioned in my opening address last year that there were two important operations that were almost unknown in the chemical manufactories of this country, I mean as far as regards the extraction of salts from solution. One was the refrigeration of liquids to produce differences in the getting out of the salts. We know these differences are important, but they have been almost, as far as I know, unutilised in this country. The other was by the application of dialysis which, though an almost unknown process in our manufactories, is a good deal used abroad, and upon that subject we shall have an important paper later on. Mr. Ellis does not treat of refrigeration from the point of view that I particularly meant. He speaks of a very slight cooling, simply 10% or so, which in this particular case makes a great difference, but sulphate of soda is a very peculiar salt, and a slight difference in range of temperature has all the advantages he mentions. I think, however, many of us—as manufacturers—will not welcome new sources of salt cake, for some of us have considerable difficulty in getting rid of what we make at present.

Meeting held Dec. 2nd, 1884.

MR. E. C. STANFORD IN THE CHAIR.

## THE CHROMATE PROCESS OF TANNING.

BY MR. W. J. A. DONALD.

THE tanning of leather is an ancient industry, perhaps as old as agriculture—the oldest of all industries; and it is bound to be an important one in all civilized countries. It is especially so in this country, whether we consider the quantity or the value of its products. Allowing two pairs of boots per annum to each individual, and assuming that each pair weighs on an average two pounds, it will be found that thirty millions of people would require 30,000 tons of leather per annum, costing over £4,000,000 sterling. But the British public require saddlery, belting, portmanteaus, carriage coverings, and hydraulic leather, the weight and value of which will probably equal that of shoe-leather. The British tanners, I am glad to find, manage, after providing for home wants, to export very largely, their export of leather and leather goods in the year 1880 having amounted in value to upwards of £3,300,000. It is not so surprising, then, to learn that the capital employed in the trade in the United Kingdom is estimated at over thirty millions sterling.



Hitherto, while Britain has maintained so high a position in the world in manufacturing leather, she has, strange to say, been indebted for a long time almost wholly to other countries for the materials with which her trade has been carried on.

Of raw hides there were imported in 1880 over 65,000 tons, valued at over £3,000,000 sterling, while virtually all the tanning material required to turn these hides into leather was got from abroad. The quantity of tanning material imported during 1879, so far as returned by the customs' authorities was in value as follows, viz. :—

Bark.....	£161,573
Sumach.....	170,692
Valonia.....	512,919
Total.....	£875,211

This sum does not apparently include bark extracts which are now so largely used in this country. If the value of these be added, the tanning material imported would be found probably to amount in value to over *one million sterling*.

While so much has been done, especially during recent years, for various trades by scientific men to provide cheap substitutes for expensive materials, and to render the British manufacture independent of materials of foreign growth, very little of a practical and lasting character has been done for the tanner in this way. Researches have no doubt been made as to the anatomy and composition of the various parts of a hide, as to the portions on which tannin and other agents have an action, and as to those others on which they have no action. Faint attempts have also been made on the basis of the knowledge thus acquired to invest tanning processes; but it is the fact, I believe, that up to this time the chromate process of tanning represents practically the only really successful attempt that has yet been made to produce leather by other tanning agents than those which have been in use since hides were first tanned. Down to the present moment, while exceptional kinds of leather, such as kids, skins, and lace leathers, have been made by tawing and shamoy processes, the staple article, such as is used for soles and uppers of boots has been wholly tanned by means of bark, bark extract, and the leaves and nuts of trees, like the oak, which contain tannic acid. By no process that I know of excepting the ordinary one, and the one I am about to refer to more particularly, has sole leather been made and rendered a marketable article.

That there is a necessity for devising new processes which shall be independent of tannic acid as obtained from vegetable sources is very evident, not only from the fact that Britain is dependent upon foreign supplies, but from the high price to which these materials have risen, their increasing scarcity, and the way in which the American and Colonial Governments are setting themselves to protect their forests from the wholesale destruction which the increasing demand for bark, and bark extracts has occasioned.

The line in which any attempts towards devising a new process has been made, has been in the direction of mineral agents. These attempts have all been based on the discovery that certain metallic salts, such as alum, sulphate of iron and bichromate of potash have the chemical property possessed by tannic acid of forming an insoluble compound with gelatine. As I have said none of these processes have resulted in any practical issue. Indeed, mock kid skins, such as are so much used for the uppers of women's boots, form really the only leather that is made by any of these processes. Sole leather and other heavy leathers as I have said could not be made thereby.

The failure to apply these processes successfully was no doubt due to the difficulty with which dense

metallic salts can be used to fill up and give body without endangering the fibre and web-like character of a hide. It seems to have been overlooked that but little of the tanning material introduced into a hide consists of tannic acid. When this was observed and taken into account by Dr. Heinzerling, the inventor of the chromate process of tanning, much was done to pave the way to success for mineral tannages.

When this process four years ago was acquired by the Eglinton Chemical Company, one of the chief producers of bichromate of potash in this country, it was in a comparatively crude state. Since then it has been developed at considerable expense, and in a thoroughly practical manner, in a small tannery which was fitted up specially for working it out. In this tannery all kinds and classes of leather have been produced from walrus hides, weighing 3cwt. each, and measuring 2 inches in thickness to the finest calf skins. The chief difficulty experienced may be said to have been to attain the requisite degree of finish or appearance while imparting substance or body, and at the same time making the leather produced heavy enough to be sold at a rate per lb. against ordinary leather. This could only be arrived at by employing skilled specialists to apply the process to the various kinds of leather. These conditions have been so fairly well reached that chromate leather is in a position to compete favourably with bark tanned leather in cost of production, while offering the advantages of being waterproof and much more durable than ordinary leather. I have had brought here to show to members a number of boots and shoes made by Glasgow boot and shoe manufacturers, wholly from the leather supplied to them from the Eglinton Company's tannery; and I think it will be admitted that they bear a remarkably close resemblance to their bark tanned rivals. The only difficulty practically which has not yet been overcome is one which is common to all waterproof leathers, viz., slipperiness on fine pavements during very wet weather; but for the million who wear nails in the soles of their boots, and for country wear, and for shooting, fishing and cricket boots it is fast being adopted as being specially suitable. I at the same time invite your attention to the sample of sole, belting and upper leathers lying before me. It will be remarked that the leather has a peculiar odour; but all leather when newly made is odoriferous, and chromate like bark leather loses much of its characteristic, and not to me unpleasant smell, in the course of a very few days after it is worn.

The chromate process of tanning was invented by Dr. Heinzerling, of Frankfort-on-Maine, in the course of an attempt to produce a cheap material for the carriage of liquids. The rapidity with which bichromate of potash penetrated a hide suggested this to him as a tanning agent. Along with it as a co-agent he selected common salt, not only because he found it gave mellowness to leather, but because it has the property of dissolving the substance in the hide called by Reimer "Coriin," the cement which binds the fine strands of the fibres together, and which stands in the way of producing soft pliable upper leather. And just as in bark-tanning, the soluble extracts from the bark are as I have said, required quite as much as the tannic-acid contents in order to fill up, and give body to the hide, so in the chromate process alumina and resinous materials are introduced after the tanning operation is finished for the same purpose. Pure paraffin wax having a high melting point has been found a very useful and convenient substance with which to treat the leather generally; but in the case of sole leather a certain proportion

of very fine colophony or common resin is added in order to give the firmness required.

The tannery equipments and the preliminary stages of preparing hides for tanning by the ordinary process apply equally to the chromate process. The tanning liquors, as I have said, consist of bichromate of potash, alum and chloride of sodium in certain proportions. The hides after being prepared in the usual way, are at first placed in a tan pit containing weak liquor just as in bark-tanning, so as to prevent the surface or grain from being drawn or wrinkled by the astringent action of the liquors. They are afterwards transferred in succession to other pits containing stronger liquors. The action of the liquor is very rapid. Heavy sole-leather hides, which ordinarily require from six to eight months, are tanned in from three to four weeks. Walrus hides, such as the sample you now see, and which usually occupy the tan pits from two to three years, are finished in three months. After the hides are taken out of the tan pits, if they are intended for sole leather, they are immersed in a solution of chloride of barium in order to neutralize any bichromate of potash which may remain in the hide, and form with it an insoluble chromate of barium. Hides intended for upper leather are similarly treated with other salts so as to render insoluble any tanning material held merely by surface absorption. The hides are afterwards passed through clean water, and then dried off to that point at which it is found convenient to introduce paraffin wax and resin. These substances are dissolved and diluted in a special manner, and so introduced as to secure that they shall penetrate and coat the minutest fibrils of the hide. After this is done the hides are subjected to much the same finishing process as when tanned by bark.

The process, it will be observed, is a rapid one—so rapid that the production of an ordinary tannery can by its means be at least quadrupled.

It is at the same time a cheap process, whether regard be had to the materials or the labour required

in its production. Materials do not cost one half the amount that ordinary tannages do. Wages for conducting the tanning process are proportionately low, while currying or dressing is performed at the ordinary piecework rates.

All this being so, it will naturally be asked what are now the obstacles to its general adoption by tanners. These have been found to be hard to entirely surmount. The chief of them all is prejudice, that stubborn foe to all improvement; but prejudice more in favour of the old rather than any well-defined objection to the new leather. Tanners, I find, are dependent upon their ordinary channels of selling, and while quite prepared to produce according to demand, are not readily disposed to manufacture what they are not quite sure they have a ready market for. Middlemen only give orders to tanners for the class of goods their regular customers require. Shoemakers as a rule cannot afford to keep a stock of chromate leather boots and shoes as well as their ordinary stock of bark tanned goods. And so it is left in the first instance to an enterprising shoemaker or to an enterprising tanner who is also a shoe manufacturer, to overcome this prejudice and introduce chromate leather to the general public. In a very short time this will necessarily take place.

In the meantime, as the testimonials circulated will show, chromate leather has already taken a good place, not only for ordinary purposes, but especially for hydraulic, belting and waterproof shoe leather. In the future, it is destined, I believe, to take a much more prominent place. For, apart from tanning in this country, the process, I think, is likely, owing to the cheapness and convenient bulk of the materials, to be adopted at the sources of supply of raw hides, in South America, India, and the Colonies. These cost nearly as much to dry and salt them for shipment as it would cost to tan them by this process, taking into account the great loss of weight and damage to the hides which take place in drying and salting.

*Extract from Report, with a Statement added showing Comparative Results.*

Number of Test.	DESCRIPTION.	DIMENSIONS OF SAMPLES.			BREAKING STRAIN.		WEIGHT.	COMPARATIVE RESULTS.	
		Breadth	Thick-ness.	Area.	Total.	Per Sq. In.		Stronger by	Weaker by
		Inch.	Inch.	Sq. In.	Tons.	Tons.	Oz. Dwt. Gr.		
573	Sample of Bark-Tanned English Bend	3.92	.24	.94	1.375	1.462	6 9 8	..	15 %
574	„ Chrome „ „ „	3.92	.22	.862	1.275	1.479	5 14 11	15 %	..
575	„ Bark „ Foreign „	3.92	.235	.921	1.1375	1.235	6 4 11	} ..	11 %
576	„ „ „ „ „	3.92	.23	.901	1.45	1.609	6 6 8		
577	„ Chrome „ „ „	3.92	.20	.784	1.6925	2.12	6 12 7	} 11 %	..
578	„ Bark „ Belting „	3.92	.195	.764	1.35	1.767	6 10 17		
579	„ „ „ „ „	3.92	.255	.999	1.55	1.551	7 19 7	} ..	31 %
580	„ „ „ „ „	3.92	.215	.842	1.5	1.78	6 7 11		
581	„ Chrome „ „ „	3.92	.23	.901	1.875	2.081	6 3 19	} 31 %	..
582	„ „ „ „ „	3.92	.25	.936	1.80	1.825	6 17 21		

\*See comparative area and weight, which proves this Sample of Chrome Tanned Leather to be strongest by 15 per cent.

The Table of Comparative Results has been made up by taking the weights of all Samples into account in the same way.



*Results of Experiments to ascertain the Tensile Strength of Six Pieces Chromate-Tanned and Six Pieces Bark-Tanned Leather—the latter being specially procured by the Subscriber to make sure of its quality being good.*

CHROMATE TANNED.		ULTIMATE STRESS.		STRESS PER INCH IN WIDTH. <i>Extension in 25 Inches.</i>				
Dimensions.	Area.	Total.	Per Square Inch.	200	400	600	800	1000
Inches.	Sq. Inch.	Lb.	Lb.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
8 by '23	2'240	7378	3297	8'48 Set	11'44	18'72 10'04	22'92	..
6 by '26	1'516	5629	3608	6'48 Set	11'28	16'84 8'16	21'92	..
5 by '25	1'250	4716	3772	9'68 Set	13'00	18'88 11'68	22'72	..
4 by '26	1'010	4364	4196	8'20 Set	13'56	16'84 9'04	20'64	23'96 11'88
3 by '25	'750	2992	3989	7'12 Set	12'68	18'68 8'72	24'16	..
2 by '22	'440	1774	4031	5'92 Set	12'52	17'96 7'92	22'20	..
Mean . . . . .		4477	3815	7'64 Set.	13'04	17'99 9'26	22'43	..
BARK TANNED.								
8 by '25	2'000	5344	2672	5'32 Set	9'28	13'68 7'92	..	..
6 by '19	1'140	3708	3252	5'80 Set	9'52	13'64 8'08	..	..
5 by '23	1'150	4459	3877	4'72 Set	7'68	11'44 5'64	15'40	..
4 by '20	'800	3283	4103	4'80 Set	7'76	11'32 5'08	15'12	..
3 by '20	'600	2156	3593	4'32 Set	7'20	9'96 5'24	..	..
2 by '21	'420	1661	3954	3'36 Set	6'08	8'40 4'48	11'12	..
Mean . . . . .		3455	3575	4'72 Set	7'92	11'41 6'07	13'88	..

As the thickness varied, each piece was measured at six places, and the dimensions given are those where fracture subsequently occurred.

NOTE.—The foregoing shows (1) that Chromate Leather excels in strength, and (2) that after it has Set under the necessary stress it still retains an extraordinary amount of elasticity, which is available for tightening machinery belting or pulleys.

#### DISCUSSION.

MR. E. C. C. STANFORD in opening the discussion, said: I don't wish in any way to point out the direction which the discussion of this paper should take, but I think it would be extremely interesting if some of those present would give us their views as to the chemistry of this process. I would ask Mr. Donald, in the first place, if he can tell us whether the effect of this is due to exposure to the light, or whether the chromic acid itself has a particular action on the skin?

MR. DONALD in reply, said: The organic matter of the hide decomposes the chromates and reduces the chromic acid into the insoluble chromic oxide. The chromic acid, at the same time, has the effect (which is promoted by the action of light) of rendering the gelatin of the hide itself insoluble. The action is mutual and simultaneous.

MR. E. C. C. STANFORD: What I particularly wanted to point out is this: Of course we all know that while bichromate of potash has no action upon gelatin in the ordinary way, yet when that mixture is exposed to the light the chromic oxide becomes insoluble, and, I understand, or at least I have understood, that that was the action effected here.

MR. MAYER, who was called on by the Chairman, said: I have certainly been over the works, as you have remarked, even at a very early stage after their

inauguration, but even at that stage the process seemed to have been worked out, perhaps not to a commercial success, but to a manufacturing success of a somewhat extraordinary character, considering the short period of time that Mr. Donald had had the experimental tannery going. I was exceedingly much interested in the matter, and did what little I could at the time to make it known beyond the city in which the tannery was established. There were some excellent results obtained in the way of the mechanical testing of the chrome leather belting at Lloyd's Proving House, Paisley Road, and I think it would be well if Mr. Donald would briefly summarize them, to show that those persons who are in the habit of using belting may find something that will serve them well, as to its physical qualities.

MR. DONALD: With reference to Mr. Mayer's remarks, and as to the tensile strength of the leather, perhaps I may be allowed to state that the hide is in its strongest condition when it is in its raw state, and that the action of tanning does not increase the strength of the hide, but rather reduces it. The shortness of the chromate process of tanning seems to me to account for the fact that both Mr. David Kirkaldy, of London, and Mr. Fraser, of Lloyd's Proving House, agree in finding that chromate tanned leather tested by them was respectively 11%, 15%, and 31% stronger than the best of bark tanned leather. Long immersion in tanning liquors, and the long time which elapses before these materials penetrate to the

centre of thick hides, combine, apparently, to dissolve and weaken the fibre of the hide.

Dr. WALLACE : Mr. Donald's paper to-night has been exceedingly interesting, but to my mind it has not been quite so full of detail as it might have been, but these details may be filled in by answers to questions. I should like to ask Mr. Donald what is the increase of weight in the hide by this process of tanning, and whether it is greater or less in the ordinary process? Then, I should like also to know what is the increase of thickness as compared with the ordinary process of tanning? The Chairman has already anticipated me in one of the questions I was about to ask, viz., whether the tanned leather contains chromic acid or oxide of chromium? I have some difficulty in forming the conclusion in my own mind, from general experience, whether it will be so or not. My own impression is that if there is decomposition into oxide of chromium it is only partial, and that part of the chromic acid remains in that condition in the finished article. But I already gather that Mr. Donald is not able to inform us upon that point. I should like to know further what is the actual proportion of oxide of chromium contained in the leather, that is, if 100 parts of it are burned what chromic oxide would be contained in the ash of it, and how much alumina there is in the finished article, for it appears to me that the alumina plays an important part in the preparation of this chromate tanned leather. With regard to belting I should like to be informed whether this article resists the force of stretching more or less than the ordinary tanned leather, for that is a point of very great importance. We all know—at least those who are in the habit of using belting in machinery—that very great trouble arises from the stretching of these belts, and that they require to be tightened up frequently, and if this leather gives way less than ordinary leather that is a decided advantage and ought to be known. I have only further to say that I have worn a pair of chrome tanned leather boots for about a year and a half, and to this day they are perfectly watertight. There is this difficulty, however, that when the Caithness pavements of our Glasgow streets are just slightly wet going over them with chrome tanned boots is a somewhat dangerous experiment, and I have frequently had to resort to the middle of the street; but whenever I have a hard day's walking in the country, in rainy weather, I put on my chrome tanned boots and know my feet will keep perfectly dry.

Mr. COLEMAN : I should like to know from Mr. Donald whether chrome tanned belting is in practical use in engineering works. It appears to me that this is one of the most important applications of the leather, and the tendency of engineers to use belting, in preference to communicating motion by wheels, and so on, is more and more evident, so that the importance of having good leather belting is increasing year by year in all engineering operations.

Mr. STANFORD : I would like to ask Mr. Donald if he considers light necessary in this process?

Mr. DONALD, in reply to the various questions, said : With reference to the Chairman's last question I can only say that in carrying out the process at the tannery special regard has not been paid to the admission of light to the pits, but it is nevertheless a fact that if chromate hides are much exposed to the light they rapidly change in colour from yellow to green, thus showing that the light has the effect of quickening the reduction of the chromates into the insoluble oxide of chromium.

Mr. STANFORD : That would deteriorate the leather, would it not?

Mr. DONALD : I don't think it would; we find, as a matter of fact, that the greener the leather the more

satisfaction it gives to the shoemaker and wearer. It does not look so well and the green colour is avoided simply because of the prejudice it creates in the minds of customers. With reference to Dr. Wallace's questions, and first, as to the matter of weight, I can only say that I believe that, by the chromate process at least, an equal yield of leather can be obtained from the raw hide to what is got by the ordinary process of tanning. What is called a market hide, that is to say a hide taken from the slaughter house, is expected to yield about one half of its weight. A dried hide gives very good results indeed if it turns out equal to its original weight. Salted hides give good results when they yield three-fourths of their original weight. The weight, however, as you can easily understand from the nature of this process, can be increased by simply charging the hides with an extra quantity of the materials which are used in the process. It will readily be understood that extra weight can only be got by increasing the thickness, and so reducing the area of the surface. Extra weight is only of importance to the tanner who has to sell by weight in competition with tanners who sell ordinary leather by weight. To the tanner who can show to his customers that he gives equal amount of surface, and who adjusts his price accordingly, shortness of weight can be of no importance. For the user whose chief consideration is to get a certain number of soles or uppers from a hide it is no loss to pay a proportionally higher price if the hide weigh less than ordinarily. And for the tanner who is at the same time a shoe maker the matter of weight is of no importance whatever. In regard to the matter of thickness I don't think that by the chromate process, as a rule, equal thickness to what can be got by the ordinary process is in practice obtained, because it would appear that by the ordinary process of tanning the longer the hide lies in the tanning liquor the surface takes up a large quantity of tannic acid and soluble extracts of the bark, and swells and enlarges very considerably, enlarges, I believe, to such an extent as to actually weaken the fibre of the hide itself, while on the other hand, on account of the greater density of the chromate tanning materials, they occupy less space, and therefore do not swell or enlarge the cellular web of a hide. With reference to the state in which the chromate salt exists in the hide, I can only repeat that it is believed to be, in course of time, reduced to the state of oxide of chromium. The greenness which you see is not surface greenness, but actually extends to the interior of the hide. Some time ago the question was raised whether the tanning material of these hides could not be dissolved out, and whether in the event of its being at all soluble it might not injure the hide. With the view to a settlement of that question I forwarded to Dr. Wallace's partner (Dr. Clark) a number of samples of leather some time ago (three or four years ago), and if you will allow me I will just read the report of his test.

*Report upon six samples of Chrome-Tanned Leather, received from the Eglinton Chemical Company, Limited, on the 19th inst.*

I have made a careful analysis of six samples of Chrome-Tanned Leather, labelled respectively, "Foreign Bend," "English Bend," "Hide Batts," "Strap Butts," "Buffalo Hide," and "Calf Skin," for the purpose of ascertaining the total quantity of chromium which they contain, and the amount which is extracted by the action of water under different circumstances. The samples were cut into pieces about two inches square, and their chromium contents



are stated for convenience in the form of bichromate of potash.

	Foreign Leather per Cent.	English Leather per Cent.	Hide Butts per Cent.	Strap Butts per Cent.	Buffalo Hides per Cent.	Calf Skin per Cent.
Total chromium contents, calculated as bichromate of potash	3.30	3.47	3.97	4.80	6.18	3.50
Quantity extracted by boiling in water for half-an-hour	.005	.018	.006	.018	.051	.006
Quantity extracted by steeping in cold water for 12 hours	.001	.019	trace	.006	.022	.060
Quantity extracted by steeping in cold water for 24 hours	.005	.027	trace	.007	.043	.077
Quantity extracted by steeping in cold water for six days	.014	.091	.025	.017	.135	.123

A sample of good bark-tanned leather obtained direct from one of our leading tanneries, when treated in a similar manner, gave the following results:—

	Per Cent.
Tanning material extracted by boiling water for half-an-hour (containing .77 of Tannic Acid)	2.13
Tanning material extracted by steeping in cold water for 12 hours (containing 1.08 of Tannic Acid)	2.99
Tanning material extracted by steeping in cold water for 24 hours (containing 1.3 of Tannic Acid)	4.45
Tanning material extracted by steeping in cold water for six days (containing 2.55 of Tannic Acid)	6.79

From the above results it will be observed that the quantity of bichromate of potash which I have been able to dissolve out of the samples of Chrome-Tanned Leather, even by steeping in water for a week, is very minute, but even this quantity, small as it is, will probably become less by the reducing action of the organic matter on the chromic acid. In any case, I am of opinion that the remaining chromium compound in the leather will not be dissolved out by any treatment to which the leather is likely to be subjected, and cannot be hurtful to the feet.

With reference to the matter of stretching I think it well to say that the extent to which a belt stretches depends entirely on the way in which the hide from which it is made is at first prepared. First-class belting butts are stretched while they are in the wet state, and if stretched in that state and kept under tension until they are perfectly dry, should attain the very minimum of stretching. The rule which applies to ordinary leather applies equally to chromate leather. At first in its manufacture it was not so carefully stretched simply because the necessity for stretching it at first was not so well known. Now it is carefully stretched in the early stage of its manufacture, and for a considerable length of time there have been no complaints on the subject of the stretching of chromate leather. The percentage of oxide of chromium present in the leather averages equal to about  $3\frac{1}{2}$  to  $3\frac{3}{4}$  of bichromate of potash. The quantity of chloride of sodium averages about  $5\frac{1}{2}$  to 6%. With reference to the extent to which leather belting for engineering purposes is in use I may say that there are very few firms in this neighbourhood who have not been supplied with chromate leather for belting or hydraulic purposes, while in England and Ireland the Eglinton Company have many regular customers. No other belting is used at the Eglinton Company's works, and whenever belting has to be exposed to the action

of the weather or to a cold damp atmosphere, in which india-rubber belting can alone be used, it gives special satisfaction. For hydraulic purposes such as for hydraulic presses, pump buckets, &c., it is used by the Admiralty, and by the leading hydraulic engineers and colliery proprietors in this country, such as Sir Joseph Whitworth & Co., Messrs. Shaw & Co., Messrs. Addie & Sons, and by the large railway companies.

Mr. STANFORD, in concluding the discussion, said: The percentage of alumina appears to me very large, but I cannot help thinking that light has a great deal to do with the effect produced, for the mixture of gelatin and bichromate of potash is so sensitive to light that even a few minutes' exposure renders it insoluble in water. I think these results which Mr. Donald has given us, through Dr. Clark, are extremely important, for we know what a large amount of tanning material can always be extracted from leather, and it appears gelatin is in a more insoluble condition in this chromate tanned leather. I may perhaps be permitted to mention, at the risk of being considered to harp too much on one subject, that the substance I have obtained from sea-weed called algin, has precisely the same effect when treated with bichromate of potash, and always becomes insoluble after a few minutes' exposure to daylight, although differing materially in other properties from gelatin. The whole subject has been fully brought before us, and I think the details that have been brought out in the discussion are also valuable.

## Obituary.

### ADOLF WILHELM HERMANN KOLBE.

ON Tuesday evening, November 25th, this great chemist ceased to breathe. His death was sudden, and was caused by failure of the heart, which, on post-mortem examination, was found to be considerably affected by fatty degeneration. Born at Ellrichhausen, near Göttingen, in 1818, he was educated at the Gymnasium and in the University of that town, where he studied chemistry under Wöhler from 1838 to 1842. From 1842 to 1845 he was the assistant of Bunsen, then Professor of Chemistry in Marburg. Conjointly with Frankland he was assistant to Sir Lyon Playfair (then Dr. Playfair), in London. In 1847 he became Editor of the *Handwörterbuch der Chemie* published in Brunswick. Kolbe was appointed Professor of Chemistry at Marburg, in 1851, and in 1865 he responded favourably to a call to take the Chemical Chair in Leipzig. As Professor of Chemistry at Leipzig University, he continued to work with honour, efficiency, and power, up to the time of his death.

In 1869 Kolbe became Editor of the *Journal für praktische Chemie* (founded by Erdmann), and latterly Professor E. von Meyer joined him as co-editor of this Journal. In conjunction with E. von Meyer the excellent Text-book of Organic Chemistry was completed in 1883, a work surpassed by none for clearness and exactness of details in the preparation of organic compounds. A second edition of his Short Text-book of Inorganic Chemistry (Brunswick, 1877) appeared this year, and an English translation, with certain additions, has been written by Professor Humpidge.

In conjunction with Frankland, Kolbe inves-

tigated the conversion of methyl cyanide and ethyl cyanide into acetic and propionic acids, and the results were communicated to the Chemical Society of London in 1847, and here occurs the first enunciation of the view that the fatty acids and benzoic acid contain alcohol radicles which may be regarded as replacing an atom of hydrogen in formic acid. Kolbe developed this view further, and verified it experimentally.

In 1847 Kolbe demonstrated that the compounds obtained from alcohols as cyanides of the radicles can be transformed into acids containing a proportion of carbon higher than the alcohols employed. In 1848, he next developed the view that the cacodyl compounds are dimethyl-arsenic derivatives. Likewise in 1848, Kolbe and Frankland isolated "methyl" (Dimethyl), and in 1849, Kolbe obtained "methyl" by the electrolysis of acetic acid. (Schorlemmer first proved that this "methyl" was dimethyl, and is in fact identical with ethane.) In 1850 Kolbe developed his views on conjugated radicles, which also were modified by Schorlemmer's researches and reasonings in after years; and in 1855 he discovered the general method of conversion of an acid into the corresponding aldehyde by means of the chloride and cyanide. On the lines above indicated further important researches followed. The existence of the secondary alcohols and the constitution of the di- and tri-carbonic acids were also foreseen by Kolbe, and he and Frankland must be regarded as the authors of the doctrine of the saturation-capacity of carbon—a doctrine the truth of which forms as it were the backbone of theoretical organic chemistry.

Kolbe rendered important service in the discovery of a new and general reaction for the preparation of oxy-benzoic acid (salicylic acid), which, after the discovery of the important antiseptic properties of that substance, proved of great technological value.

His family life had been very happy; he was, as a teacher, most highly esteemed both by his assistants and by his pupils, and as a man he was noted for his generosity. In later years he had become remarkable as a severe and unsparing, if not merciless critic. The most decided and powerful opponent of the type- and structural-theories, he not only waged continual warfare with the founders and representatives of these theories, but he even attacked them with regard to their use of their mother tongue; and it must be confessed in his strictures and satires he mingled sufficiently gall and vinegar, to suggest at times, that the cogency of the increasing experimental evidences supporting these newer and to him obnoxious theories, was being slowly driven home. But let us look again at the brighter side of this strong, although somewhat rugged nature; it was also a nature both high-strung and sensitive. On the day of his death Kolbe had been working in his laboratory till five o'clock in the evening, and had attended a meeting till eight o'clock, and it was on the very threshold of his house that he fell. He died as men of his kind would wish to die—in harness.

It may be a fact of some interest that one of our members, Dr. Gerland, was the first graduate for the degree of Doctor of Philosophy under Kolbe in Marburg, (1853,) and that Dr. Peter Griess, a vice-president of this Society, first discovered the diazo-compounds in his laboratory in 1862.—W. S.

## AUGUSTUS VOELCKER, Ph.D., F.R.S.

A MEMBER OF THE SOCIETY OF CHEMICAL INDUSTRY.

ON Dec. 5th, at his residence in Kensington, this authority, one of the greatest in the country, on agricultural chemistry, departed this life. He was born at Frankfort-on-the-Maine in 1823, and received his chief education at the University of Göttingen, and in early life came to England. He was assistant to the late Professor Johnston, of Edinburgh, in 1849, and was appointed Professor of Chemistry in the Royal Agricultural College of Cirencester in 1852, which office he held till 1862, when he became Professor of Chemistry to the Royal Agricultural Society of England. He was the author of several works, as the "Chemistry of Food," "Chemistry of Manures," "Lectures on Agricultural Chemistry," etc.—W. S.

## Journal and Patent Literature.

### II.—FUEL AND LIGHT.

A. Proposal to Obtain a Constant Illuminating Unit. II. v. Hefner-Alteneck. Chem. Centr. Bl. 1884, 661.

THE term "illuminating unit" is said to denote the illuminating power of a free-burning flame ascending from a solid wick, which is saturated with amyl acetate, and fills up the entire space of an annular white metal tube. The latter is 25mm. long, and has an internal diameter of 8mm., and an external diameter of 8.3mm. The height of the flame from the edge of the tube to the top of the flame is 40mm., and the measurements are taken at least 10 minutes after the lamp has been lighted. Having prepared a lamp in accordance with these directions, the authors made the following tests:—

Substance used.	Formula.	Percentage weight of Carbon.	Boiling Point.	Illuminating Power.	Combustion of 1 gram. in seconds.	Grams of Carbon burnt in 100 seconds.
1	2	3	4	5	6	7
Amyl valerate....	$C_{10}H_{20}O_2$	69.7	195°	1.03	420	0.162
„ acetate ....	$C_7H_{14}O_2$	64.6	135°	1.00	388	0.166
„ commercial	—	—	—	1.00	—	—
Amyl formate ....	$C_6H_{12}O_2$	62.1	122°	1.01	372	0.163
Isobutyl acetate ..	$C_6H_{12}O_2$	62.1	116°	0.99	373	0.163
„ formate..	$C_5H_{10}O_2$	58.8	98°	0.97	355	0.166
Ethyl acetate ....	$C_4H_8O_2$	54.5	75°	1.24	212	0.285

Columns 5, 6, and 7, show the interesting behaviour of the above substances (with the striking exception of the last) that their consumption on burning with equal sized flames and nearly equal illuminating power is different, so that the quantities of carbon consumed in equal spaces of time is again approximately the same. Of all the substances employed amylacetate seems the most suitable for the unit lamp. The use of commercial amyl acetate instead of the chemically pure, the preparation of the wick of threads, each 1m. m. in diameter, or cutting the wick in a rounded summit about 2m. m. high instead of flat, gave results differing very little from each other, so that the reproduction of this unit of light would present no difficulties.—D. B.



### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Antiseptic Treatment of Timber.* S. B. Boulton, Assoc. Inst., C.E. Minutes of Proceedings. Institution of Civil Engineers. May, 1884.

THE author, in this contribution to our knowledge of this subject, gives a short account of the history and development of the use of antiseptics for preventing the decay of timber, together with some results derived from his large experience in connection therewith. In referring to the theories as to the causes of putrefaction, the author states that it was not until the commencement of the present century that putrefaction, at least of vegetable matter, began to be generally recognized as a species of fermentation, although at first it was not even then admitted that ferments of any kind were the products of living organisms. Many of the antiseptics proposed by the early investigators, particularly the alkalis were found to be absolutely injurious to timber, and although towards the close of the last century and the beginning of the present, experiment was greatly stimulated by the wants of the British navy, it was not until the introduction and growth of the railway system that the antiseptic treatment of timber made rapid progress. By the year 1838 four processes for prolonging the duration of railway sleepers were competing with each other. These were:—(1) corrosive sublimate, introduced by Kyan; (2) copper sulphate by Margary; (3) zinc chloride by Burnett, and (4) heavy oil of tar (afterwards called creosote) by Bethell. Kyanizing was for a long time by far the most popular of the timber preserving processes in England. Used in sea-water, however, by the British Admiralty, it proved a failure, although in comparatively dry situations the process has met with a considerable amount of success. The copper sulphate process was practised largely in France, long after it had been given up in England. It is still in use in France to a limited extent for sleepers and telegraph poles. The process of Burnettizing was at one time much patronized by the British Admiralty. For railway sleepers it was formerly extensively used in France. It is still adopted in Holland and Germany. Chloride of zinc is a powerful antiseptic, but its weak point for wood preserving consists in its extreme solubility in water. The practical introduction of the "creosoting" process is due to Bethell, whose patent, dated July 1838, contains a long list of substances, and among them is mentioned a mixture consisting of coal tar, thinned with dead oil distilled from coal tar. It was claimed for all four of these processes that they coagulated the albumen contained in the sap of the wood, and that they formed insoluble compounds therein, thus arresting decay. Prolonged experience has, however, proved, that the metallic salts are not so permanent in their effects as the tar oils, and gradually the creosoting process took the place of the others, until in England it entirely extinguished its rivals. The author's last experience of Kyanizing in England was carried out in 1863. The experiments of Forestier, Crepin, and others confirmed the experience of English engineers as to the success of the creosoting process against the ravages of marine insects. With regard to the process of manufacture by which the tar oils used for creosoting are procured, reference is made to the accompanying table, which shows in a comprehensive form the principal substances obtained from coal tar, as it is carbonized at the gas works. These useful substances are prepared by subjecting coal tar to fractional distillation. Fig. 1 shows the usual form of the wrought iron tar still, heated by a furnace placed beneath the still, the operation being aided by the injection of steam or the application of an exhausting apparatus. The temperature during the distillation ranges from 82° to 400°. This preliminary process, although now carried out with more skill and economy than formerly, has not varied much during the last 50 years in its main object, which is to break up the tar into 3 groups of products, viz., oils lighter than water (crude naphthalas); oils heavier than water; and pitch, the residuum of distillation. The heavy oils of tar or dead oils heavier than water, con-

stitute the creosote of the timber yards. They contain numerous liquid and semi-solid substances, varying considerably in their properties, but most of them are now recognized as antiseptics. The dead oils made in London and in all places where the tar is produced from the carbonisation of the coal of the Newcastle district, are as compared with other dead oils the richest in semi-solid substances (naphthalene, anthracene, pyrene, etc.), and they require a higher temperature to volatilise. They are generally called "London oils." The oils of the Midland districts are lighter, thinner, more volatile, and contain usually a larger proportion of tar acids. They are called "Country oils." The "Scotch oils" are many of them, still lighter, thinner and more volatile; sometimes lighter than water. Referring to the question of thick and thin oils, the author proves that there is no doubt as to the opinion and practice of the earlier introducers of the creosoting process. When the construction of railways commenced in India, in 1850, the experiment was tried of forwarding creosoted Baltic timber from England. The success of creosoted timber in India was fully set forth by Bryce McMaster in two papers on Indian permanent way materials, one published in 1859, and the second in 1863, by Juland Danvers in his annual report to the Secretary of State for India for the year 1863, and in the printed report of the East Indian Railway Company for the year 1867. The author is well acquainted with the origin of all the creosote used for these earlier Indian sleepers, as his firm for many years creosoted almost all the sleepers sent abroad. He states that by far the greater bulk was "London oil," up to 1863, comparatively small percentages of "Country oil" and in some years none at all being used. At present, however, not only is a larger quantity of creosote injected, but more care is also expended in the selection of the wood than was formerly the case. If, therefore, the earlier sleepers shipped to India behaved well, it might be assumed that the quality of the creosote at least was suited to the climate. Such creosote, however, as was then used would now be rejected under the requirements of many of the specifications at present in force for the preparation of timber for tropical countries, and the author submits that it is a question for grave consideration whether the change has been for the better. It is a matter of notoriety that for many years an increasing demand has arisen for the thinner and lighter creosotes. This view was adopted by Letheby, who was further influenced by the growing recognition of the antiseptic properties of carbolic acid. In 1865 Letheby drew up a specification for an Indian railway, which contained the following stipulations: The creosote is to have a sp. gr. as near 1.050 as possible, ranging from 1.045 to 1.055. It is not to deposit naphthalene, or para-naphthalene, at a temperature of 40° F. It is to contain 5 per cent. crude carbolic acid and other coal tar acids, and to yield 90 per cent. liquid oil when distilled from its boiling point to a temperature of 600° F. This specification has long ceased to be used, but its stipulations have been copied, and in some cases carried to greater lengths, in more modern specifications, 10 per cent. of tar acids being occasionally required. Such specifications exclude the "London oils," if taken in their entirety as they come from the still. In 1848, de Gemini, in France, showed that timber cannot be permanently preserved by the use of antiseptics which are themselves soluble in water, and for that reason he preferred the use of heavy oils or bituminous substances. Rottier, in 1862, in Belgium, published the results of some experiments as to the effects upon timber of the various constituents of coal tar oil. He concluded that although carbolic acid was a very energetic antiseptic, yet that owing to its volatility the durable success of the creosoting was not due to its agency but to the heavier and less volatile oils. In 1866, Coisne, an engineer in the service of the Belgian government, made a series of experiments with the view to determine in a practical manner which portions of the tar oils best preserved timber. He procured samples of creosote from England, Scotland, Belgium and France. Four of these samples contained respectively 15 per cent., 15 per cent., 8 per cent., and 7 per cent. tar acids. The fifth was an oil of heavy specific gravity specially prepared and containing no tar

acids. Yet this last sample produced better results than any of the others. Each sample was divided into portions. Wood shavings were saturated with these oils in the following different ways:—(1) with the creosotes as received; (2) with the creosotes supplemented by additional quantities of tar acids; (3) with the creosotes supplemented by some of the heavier portions of the same oils distilling over at a temperature exceeding  $320^{\circ}$ ; (4) with the original creosotes divided into the lightest, the medium, and the heaviest portions. With each of which the shavings were separately saturated. A putrefying pit (*pourrissoir*) was prepared, in which the shavings were placed on the 10th November, 1866, together with other shavings not prepared. On the 16th November, 1870, they were removed and examined. The results were strikingly in favour of the heavier oils, and adverse to the tar acids. The shavings prepared with the lightest portion of the oils, although they contained the largest proportion of tar acids, were in the worst condition. Those prepared with the oils somewhat heavier were in most cases better preserved. Best of all were the shavings prepared with the heaviest oils containing no tar acids; these were all perfectly sound. The unprepared shavings were all rotten. These experiments are recorded at length in the *Annales des Travaux Publics de Belgique*. Their results have considerably influenced the practice of railway engineers on the Continent. The specification of the Belgian Government does not stipulate for any tar acids. It requires that at least two-thirds of the creosote must have been obtained by distillation at a temperature exceeding  $250^{\circ}$ , and that nothings should distil under  $200^{\circ}$ . It allows of 30 per cent. of naphthalene, calculated at the ordinary temperature. The experiments of de Gemini, Rottier and Coisne appear to be in absolute contradiction with the theory that the creosoting process owes its success to the tar acids. Yet the fact cannot be doubted, that the tar acids are powerful antiseptics, and that their presence arrests decay. Authorities on antiseptics agree as to the following facts: That carbolic acid is volatile at ordinary temperatures. That it is soluble in water. That its combinations are not stable. That it is a powerful germicide, but that its efficacy ceases as soon as it evaporates or is washed out of the substances intended to be preserved. If these facts be correct, can carbolic acid be considered as a durable agent among the oils injected into railway sleepers? Especially can this be the case in those tropical countries, where extreme heat or torrential rains or alternations of both are prevalent? In 1867, Coisne obtained some creosoted sleepers which had successfully resisted decay during periods of from 18 to 20 years. On examination no tar acids could be detected, but the presence of naphthalene and an oil, which did not boil under  $230^{\circ}$ , was established. In 1882, the author caused some similar experiments to be made. He obtained 11 pieces of old creosoted sleepers from the authorities of the London and North Western Railway. They had been in use for the following periods: 1 specimen 16 years, 1 ditto 17 years, 2 ditto 20 years, 2 ditto 22 years, 1 ditto 28 years, 2 ditto 29 years, 1 ditto 30 years, and 1 ditto 32 years. Sleepers were also received from the Taff Vale Railway, the South Eastern, and Great Eastern Railways, which had been in use for periods varying from 14 to 23 years. A portion was also taken from a creosoted pale fence, which had been fixed in the Victoria Docks in 1855, and which is still in place, perfectly sound and strong after 29 years' use. A careful analysis of these 17 specimens, all of ordinary Baltic fir, gave the following results:—(1) In no cases were any tar acids detected by the ordinary test. (2) In 14 out of 17 specimens the semi-solid constituents of the tar oils were present; in 12 of them was naphthalene, this body being in some cases in considerable quantity. (3) Only small percentages remained of oils distilling below  $230^{\circ}$ . In the majority of instances from 60 to 75 per cent. of the total bulk of substances retained in the wood did not distil until after a temperature of  $315^{\circ}$  was reached. It is clear, therefore, that these timbers had been preserved by the action of the heaviest and most solid portions of the tar oils and that the other constituents had disappeared. (4) In some of these specimens acridine was detected. This is probably

the first occasion on which acridine has been publicly mentioned in connection with the injection of wood, and the author is of opinion, that it will come to be recognized as one of the most valuable constituents of the tar oils for timber preserving purposes. Portions of the same specimens of wood were tested by Greville Williams. By employing the extremely delicate bromine and ammonia test, used by Cloetta and Schaar for detecting traces of phenol in urine, he in most cases succeeded in detecting minute quantities of carbolic acid, which, however, were too small to account for the preservation of the timber. In all specimens, save two, naphthalene was found. The organic alkaloids which remained were sufficient to allow of quantitative estimation. Greville Williams concludes, that the preservative action of the creosote oils is due more to the alkaloids than to the tar acids, as the former remain after the latter have disappeared. The following experiments are quoted by the author, as being in strict accordance with the properties which characterise naphthalene and carbolic acid:—(1) If tar acids and naphthalene be separately exposed, either at the ordinary temperature, or at the tropical heat of  $54.4^{\circ}$ , the tar acids will evaporate with much more rapidity than naphthalene. (2) Injected into timber the same results follow. (3) Light thin oils, containing large percentages of tar acids, evaporate more quickly than heavier oils, containing less tar acids and more naphthalene, when tested by methods Nos. (1) and (2). (4) By repeated washings with cold water, all the carbolic acid and all, or nearly all, the cresylic acid can be washed out, both from Country and London oils, as illustrated by the following:—

*Experiment with two specimens of tar-oils containing respectively 10 per cent. and 17.5 per cent. of tar-acids by the ordinary caustic soda test. The object was to ascertain what proportion of the tar-acids could be removed by repeated washing with cold water.*

SPECIMEN OF MIXED LONDON AND COUNTRY OIL.			SPECIMEN OF COUNTRY OIL.		
<i>Test before Experiment.</i>			<i>Test before Experiment.</i>		
		Per Cent.			Per Cent.
Total distillate at $600^{\circ}$			Total distillate at $600^{\circ}$		
Fahr. .. ..	70		Fahr. .. ..	85.0	
Tar-acids .. ..	10		Tar-acids .. ..	17.5	
Water .. ..	2		Water .. ..	3.0	
Sp. gr. at $60^{\circ}$ Fahr. ..	1.065		Sp. gr. at $60^{\circ}$ Fahr. ..	1.046	
20 ozs. of oil washed with cold water seventeen times (1,020 ozs. of water used).			20 ozs. of oil washed with cold water seventeen times (1,020 ozs. of water used).		
<i>Test after Experiment.</i>			<i>Test after Experiment.</i>		
Total distillate at $600^{\circ}$			Total distillate at $600^{\circ}$		
Fahr. .. ..	70.0		Fahr. .. ..	83	
Tar-acids .. ..	3.5		Tar-acids .. ..	6	
Water .. ..	1.0		Water .. ..	3	
Sp. gr. at $60^{\circ}$ Fahr. ..	1.071		Sp. gr. at $60^{\circ}$ Fahr. ..	1.049	
Again washed with cold water fifteen times (900 ozs. of water used).			Again washed with cold water fifteen times (900 ozs. of water used).		
<i>Test after Experiment.</i>			<i>Test after Experiment.</i>		
Total distillate at $600^{\circ}$			Total distillate at $600^{\circ}$		
Fahr. .. ..	69.0		Fahr. .. ..	79.0	
Tar-acids .. ..	1.5		Tar-acids .. ..	3.5	
Water .. ..	Free		Water .. ..	Free	
Sp. gr. at $60^{\circ}$ Fahr. ..	1.073		Sp. gr. at $60^{\circ}$ Fahr. ..	1.053	

The small proportion of tar acid remaining, in both cases, contained no carbolic acid when examined by the bromine and ammonia test; it did not boil until  $420^{\circ}$  Fahrenheit, which may be considered as too high a temperature even for cresylic acid. It is probably one of the higher phenols. These experiments assume especial importance in considering the durable effects of various kinds of creosote for protecting timber immersed in sea water from the attacks of marine insects. Tidy has injected pieces of wood with naphthalene, and exposed them to a temperature of  $65.5^{\circ}$ . He found that the evaporation was only superficial, and that it practically ceased after 48 hours, the naphthalene below the surface remaining within the pores of the wood. Naphthalene is now recognised as an antiseptic, not so powerful in its immediate effects as carbolic acid, but more durable. In summing up the evidence which the author has produced, he asks, can the conclusion be resisted, that for the purpose



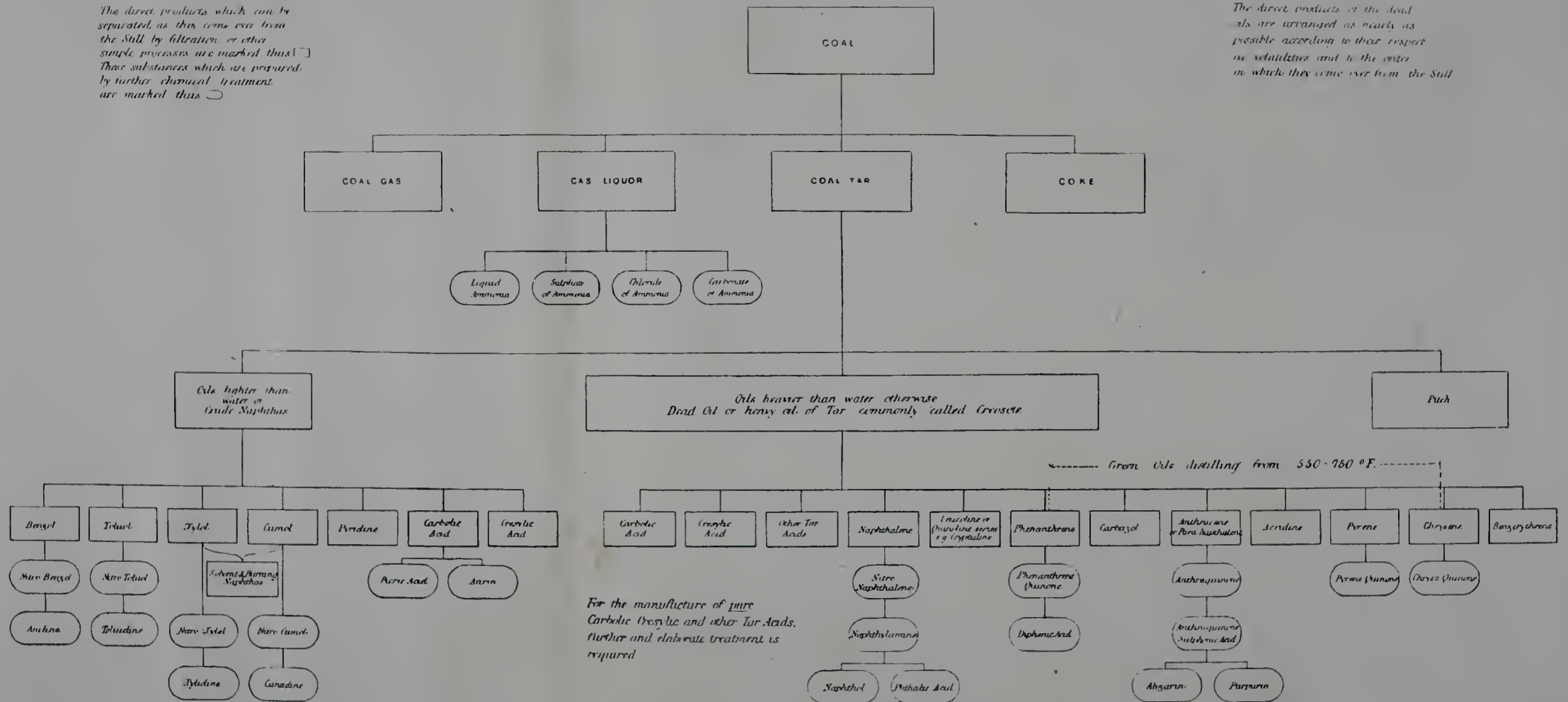
of creosoting, the efficacy of the tar acids has been overrated, and this at the expense of the more stable and enduring portions of the tar oils? The London oils, as they come from the still, are too heavy to meet the wants of the modern creosote specifications. They contain, as a rule, not more than from four to seven per cent. of tar acids, and do not yield 90 per cent. of their bulk by distillation below 315°. Therefore, a pressure is put upon the manufacturer to meet this demand by "taking out" some of the heavier portions. Thus the bulk is rendered lighter, and the proportion of tar acids to the diminished bulk is increased. But in the author's judgment, the efficacy of the oils is thereby diminished. He hopes that at least the lighter portions of the tar acids, and especially carbolic acid, may soon be relegated altogether to their important functions as sanitary antiseptics, for which they are so valuable, instead of being wasted by the attempt to use them as antiseptics for timber, for which their peculiar properties render them unreliable. Upon the whole it

would be wiser to revert to a larger extent, and with increased knowledge, to the plan of using the London oils mixed with the country oils, and encouraging, instead of discouraging, the use of the heavier portions of these oils. In 1881, Abel and Tidy drew up a joint creosoting specification in which they resolved to exclude no semi-solid substances which completely melted at 100° F. They also changed the standard of volatility from 90 per cent. at 600° F. to 75 per cent. Subsequently, Tidy not only withdrew the clause limiting to 25 per cent. the oils distilling at a higher point than 600° F., but required that at least 25 per cent. of these heavier oils must be present. Abel has recently recommended a reduction in the percentage of tar acids, and agrees with Tidy in not merely tolerating, but insisting upon, the presence in considerable volume of the heavier and least volatile portions of the oils, *i.e.*, those distilling at or above 600° F. The following are selections from the very numerous specifications which have been recommended for the preservation of timber:—

## TIMBER PRESERVING SPECIFICATIONS.

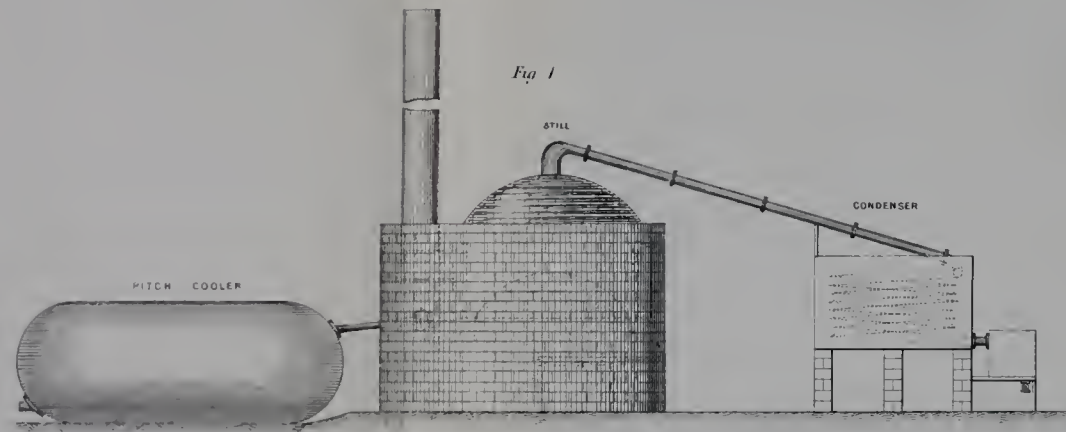
YEAR.	NAME.	SUBSTANCE EMPLOYED.	SPECIFICATION.
1832 1836	{ J. H. Kyan, } { Patent No. 6253 } { " " 7001 }	{ Di-chloride of } { mercury, or cor- } { rosive sublimate }	{ 1lb. of salt to 5 gallons of } { water, used hot or cold. Im- } { mersion for from seven to } { twenty-one days. }
1837	{ J. J. L. Mar- } { gary, Patent } { No. 7511 .... }	{ Sulphate or acetate } { of copper ..... }	{ 1lb. of salt to 4 gallons of } { water. Immersion two days } { per inch of thickness of } { wood. }
1838	{ Sir W. Burnett, } { Patent No. } { 7747 ..... }	Chloride of zinc .....	{ 1lb. of salt, to 5 gallons. Im- } { mersion for from ten to } { twenty-one days. }
1838	{ J. Bethell, } { Patent No. } { 7731 ..... }	{ Saline solutions and } { resinous or bitu- } { minous substances. } { Coal tar thinned } { with one-third to } { one-half its own } { quantity of dead } { oil. Caoutchouc } { or resin may be } { added. }	
1865	Dr. Letheby .....	Creosote oil .....	{ Sp. gr. at 60° F. 1045—1055, as } { near 1050 as possible. Not to } { deposit naphthalene or para- } { naphthalene at 40° F. To } { yield not less than 5 per cent. } { tar-acids, and not less than } { 90 per cent. of liquid oil at } { 600° F. }
1869- 1884	{ Belgian } { Government }	Coal tar creosote .....	{ Not to contain more than 30 per } { cent. naphthalene at ordinary } { temperature. Two-thirds } { must distil above 482° F. None } { to distil under 392° F. }
1883	Dr. Tidy .....	" " .....	{ To be completely liquid at a } { temperature of 100° F. Must } { contain at least 25 per cent. } { of constituents that do not } { distil at a temperature of } { 600° F. To yield a total of } { 8 per cent. of tar acids. }
1884	{ Sir Frederick } { Abel ..... }	{ Creosoting liquor or } { heavy oil tar ..... }	{ Completely fluid at 100° F. } { To contain not less than 20, } { nor more than 30 per cent. } { of constituents that do not } { distil at a temperature of } { 600° F. To yield not less } { than 9 per cent. of tar acids. } { Spec. gravity 1.035 to 1.065. }
1864 to 1881	Cie. des Chemins de Fer de l'Ouest (France) .....	Creosote .....	{ To contain 5 per cent. of tar- } { acids (acide phénique). }

TABLE  
SHOWING THE MOST IMPORTANT OF THE PRODUCTS DERIVED FROM NEWCASTLE COAL WHEN CARBONIZED BY THE USUAL METHOD FOR THE MANUFACTURE OF GAS

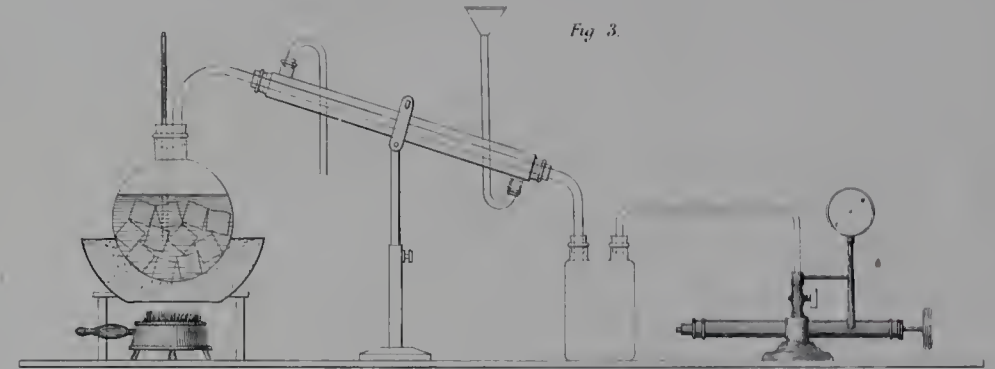




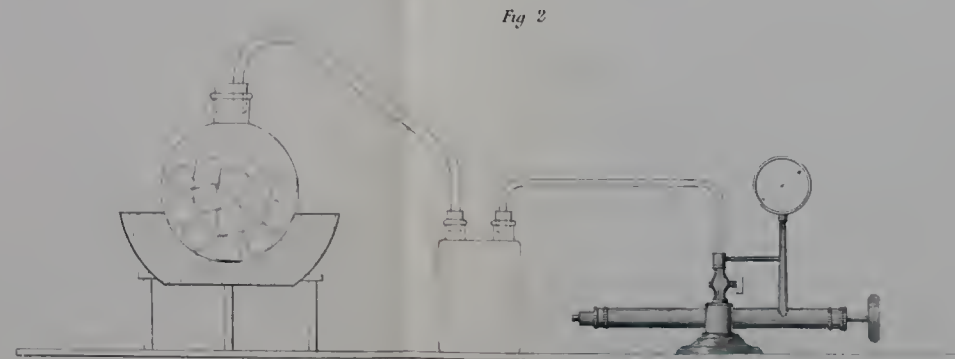
# THE ANTISEPTIC TREATMENT OF TIMBER.



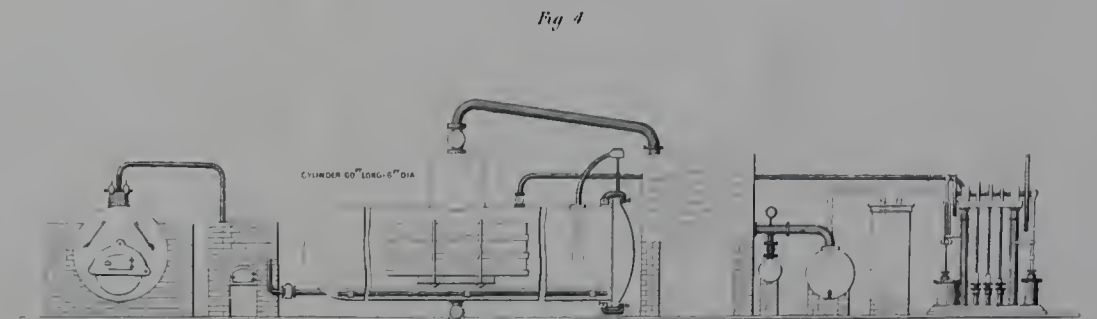
APPARATUS FOR COAL TAR DISTILLATION



EXPERIMENT



EXPERIMENT



IMPROVED TIMBER PRESERVING APPARATUS

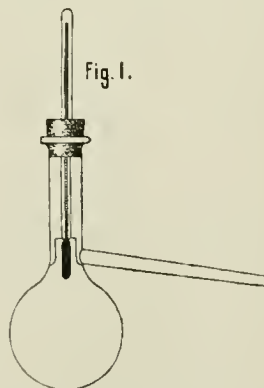
The author then proceeds to describe the conflicting theories on putrefaction down to the present period. He considers that the germ theory supplies a severe but salutary test in choosing antiseptics for the preparation of timber, and that reliance must not for this purpose be placed on antiseptics, however potent, which will readily volatilise in air or dissolve in water. In discussing the various forms of timber-preserving apparatus, it is stated that the iron cylinder and the process by vacuum and pressure, as described by Burt in 1853, is, in its main features, still in use at the present time. The hygroscopic condition of timber at the time of injection is an important element in the success of the operation, especially with the creosoting process. Neglect on this point has often been the cause of partial or total failure. The author has recently devised a method by which to get rid of the moisture in wood as part of the timber-preserving process, and without injury to the wood. Fig. 2 represents a glass flask, in which are placed some pieces of wood saturated with water, the neck of the flask being connected with an air-pump. By working the pump the air is extracted from the pores of the timber; but, however efficient the vacuum may be, no perceptible moisture is withdrawn, nor would the water be removed from the wood except by a slow evaporation prolonged beyond practical limits. This represents the ordinary action of the air-pump on timber in a creosoting cylinder. If sufficient heat be now applied beneath the flask, the water will become volatilised, and will be withdrawn rapidly in the shape of steam by the action of the air pump. But the wood will be found to crack, and open to an extent which is not desirable. This represents the result of applying dry heat. Fig. 3 represents a similar flask with a condensing apparatus added, the wet timber being submerged in creosote oil contained in the flask. The creosote is then heated to 100°, and the air-pump put into operation. The heat being communicated through an oily medium will not injure the timber from which the water is volatilised; thus the water is speedily and effectually removed and the creosote takes its place. Fig. 4 represents the ordinary creosoting apparatus supplemented by additional fittings required to carry out this process. By the ordinary method after the timber has been placed in the cylinder and the air-tight door closed, the air is exhausted; the creosote is then introduced, heated to a temperature of from 38° to 49°, when the air-pump ceases to work, and the pressure-pump is put into operation. In the new process a large dome is placed on the top of the cylinder, to which the exhaust pipe of the air-pump is attached. The exhausting process is continued after the creosote has been introduced into the cylinder. The creosote during this part of the operation should not be allowed to rise quite to the top of the vessel, a free space being preserved, and the dome kept empty so that the creosote is not drawn through the exhaust pipe. The creosote is raised to a temperature somewhat exceeding 100°, instead of 49° as heretofore. The exhausting process is continued until all the water is extracted from the timber in the form of vapour, drawn through the dome, condensed by passing through the worm of the condensing apparatus, and collected in the receiving tank, where the quantity extracted can be measured. With charges of very wet sleepers the author has succeeded in withdrawing water equal in volume to 50 gallons per load of timber (50 cubic feet), and replacing this water with an equal volume of creosote. A slight additional cost, and a few hours' additional time are necessary for dealing with very wet timber by this process as compared with the ordinary method. But the expenditure in time and money is not so great as would be required by stoving the wood before creosoting. If in the absence of artificial methods, timber be stacked for six months, as it should be, the interest on capital represents a certain expenditure also. In the discussion on this paper a number of eminent chemists and engineers took part, and there appeared to be a general consensus of opinion as to the value of the creosoting process when properly carried out, and of its superiority over the other processes. The author, in his reply, was

able to produce additional evidence upon points raised during the discussion. He again referred to the extreme importance of all timber being deprived of moisture as an essential preliminary to the injection of the antiseptic oils. He quoted the evidence and experiments of a large number of authorities (including Angus Smith, Sanson, Koch, Boillat, Tidy, Greville Williams and others) as to the volatility of the tar acids and the instability of their combinations with albumen. He further alluded to the considerable amount of practical evidence which had been adduced during the discussion as to the complete success of heavy creosotes of which the composition had been recorded, and which contained but small percentages of tar acids, whilst on the other hand it had been proved in numerous instances that carbolic acid had had no permanent effect on timber owing to its instability. The more enduring portions of the creosote oils containing germicides and solidifying materials, and which would not evaporate except at very high temperatures were, in his judgment and experience, the most valuable and reliable antiseptics for the treatment of timber.

During the discussion there were exhibited numerous specimens of successful creosoted timber of very long duration, the analyses of the oils, which they were found to contain, having been referred to in the paper; also a collection of products derived from coal tar to illustrate the accompanying table. The appendix to the paper contains 162 references to various authorities, a condensed translation from "Koch" on Disinfection, and one from a paper by Boillat on antiseptics, together with other documents confirming the view of the author.—D. B.

*Fractional Distillation in Relation to Technology.* G. Lunge. *Dingl. J.* 254, 70. From *Chem. Ind.* 1884, 150.

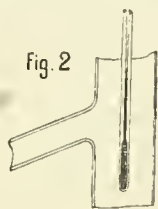
The Commission appointed by the *Verein für Chem. Ind.* to prepare a scheme of standard methods of analyzing, and reporting upon chemical products, considered this subject only in regard to compounds of the fatty series. The author, recognising that it is of much greater importance in relation to the benzene hydrocarbons and the aromatic compounds generally, has pursued the inquiry in this direction. It was found from the replies to questions addressed to manufacturers that in nearly all cases glass vessels are used, the exceptions being only two in number, and in these, vessels of copper were employed. With regard to the form of the vessel, in two cases only was the retort employed as in this country, and the author strongly recommends its discontinuance here, in favour of the fractionating flask,



which is almost exclusively employed in Germany. In the majority of cases the simple flask is used, and in these the operation requires to be many times repeated in order to effect a separation approaching completeness, e.g., of benzene and toluene. The addition of the Hempel tube, in which the vapours are made to pass through a column of glass beads, very considerably expedites the fractionation, and is to be preferred before all other "dephlegmation" apparatus hitherto devised. In two cases, on the other hand, the flask represented in fig. 1



is employed, with the obvious purpose of effecting the most rapid removal of the vapours and of preventing condensation within the flask. With this flask thermometers are used, in which the graduation corresponding to the particular boiling temperature of the liquid which it is required to separate, is situated just above the cork, so that the mercury column is entirely immersed in vapour. This method is to be recommended in cases where it is desired to isolate a single liquid, but not for serial fractionations. For such the author recommends a method which has been practised for some time in his laboratory, which consists in leading the vapours through a tube inclined upwards and surrounded by a jacket heated to the boiling temperature of the liquid to be isolated, at which it is maintained till nothing more distils. The temperature of the jacket is then raised, and the distillation continued at the next constant. From this tube the vapours are taken through an ordinary condenser, as scarcely requires to be said. Distillation combined with observations of temperature

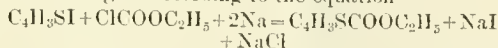


is dependent upon so many variables, that too much care cannot be taken in controlling the results. The most complete control is afforded by the distillation of a chemically pure specimen under conditions identical with those which will obtain in the operation with crude or mixed products. With regard to thermometers, the mercury bulb should not exceed 10mm. in length, and should be situated as represented in fig. 2. Barometric observations and corrections for the observed variations are highly essential. In cases, however, where the constants have not been determined with accuracy for any given liquid, the simultaneous observation of the boiling point of a pure specimen will serve. In general terms care must be taken to maintain the conditions of distillation rigidly constant—neither the inclination of the condenser, nor even the length and diameter of the tube, are to be neglected. Special precautions are given to be observed in the distillation of various liquids.

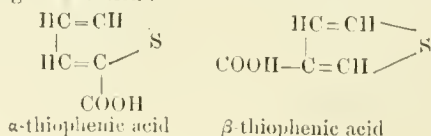
—C. F. C.

***β*-Thiophenic Acid.** R. NahnSEN. *Berichte der deutsch. Chem. Gesellschaft*, 17, 2192.

This acid is obtained by the method of Wurtz, *i.e.*, by acting on thiopheniodide with chlorcarbonic-ether and sodium amalgam according to the equation



With 50grms. of thiopheniodide the reaction is complete in about an hour. The cooled mass is then separated from the mercury by filtration and distilled with steam. The ether of *β*-thiophenic acid distils over as an oil, together with a quantity of a crystalline substance which has not been further investigated. Thiophenic ether is taken up with ether and saponified with concentrated potash solution. The oily portion, chiefly thiopheniodide, is then separated and the aqueous solution acidified with sulphuric acid and shaken with ether. On evaporation of the ether the new acid crystallizes out. This acid (m.p. 129°) is isomeric with that of V. Meyer and H. Kreis (m.p. 118°) and, according to V. Meyer's formula for thiophen, the acids will probably have the following constitution:



*β*-Thiophenic acid is similar in its properties to benzoic acid and distils without decomposition at 260°. With  $PCl_5$  it forms thiophenic chloride, which, on treating with ammonium carbonate, is converted into an amide. The author has also obtained from *β*-thiophenic acid a nitro-compound.—J. B. C.

***On the Estimation of the Three Xylenes in Coal Tar.*** By A. Reuter. *Ber. Chem. Ges.* 1884, p. 2028.

The author expresses his doubt as to the reliability of Levinstein's method for the estimation of the three isomeric xylenes in coal-tar. He finds that with very dilute nitric acid the isomers cannot be completely separated from metaxylene, while more concentrated acid also attacks the two other xylenes. Concentrated sulphuric acid behaves in a similar manner, paraxylene being acted upon even by an acid of ordinary strength, although not as readily as its isomers. If the crude xylenes contain paraffin, the capability of the isomers to resist the action of the sulphuric acid is also considerably increased. The last traces of xylenes can be extracted from the paraffin only by using a great excess of fuming sulphuric acid.—F. M.

***On the Pyrogenic Formation of Diquinoline.*** By J. Zimmermann and A. Müller. *Ber. Chem. Ges.* 1884, p. 1965.

By passing quinoline, prepared by Skraup's method, through an iron tube heated to a dull red heat a dark brown or almost black oily liquid is obtained from which, by fractional distillation and recrystallization of the part distilling above 360°, a compound crystallizing in scales was isolated. This was found to have a composition corresponding to the formula of a diquinoline  $C_{18}H_{12}N_2$ . It is identical with the base described by Japp and Graham as diquinolyline.—F. M.

***Analysis of Coal-Tar Products.*** *Die Chem. Industrie* 7, 311.

At the general meeting of the German Society of Chemical Industry, held at Dresden, September 14th and 15th, Dr. Lunge read the report of the commission appointed for the consideration of the best methods of analysing and determining the value of chemical products. The following methods are mentioned as being generally employed in the analysis of coal-tar products:—Benzene is always examined by fractional distillation. For creosote oil Rütger's test alone is used (Wagner's Jahresbericht 1868, p. 713). It is probable that this simple test would be considerably improved by defining its conditions more accurately, *e.g.*, by always employing caustic soda solution of fixed strength for the extraction of the acids. The precipitation of phenol as tribromophenol should not be employed for the valuation of commercial carbolic acid, because the higher homologues of phenol also give tribromophenol, their  $CH_3$ -groups being oxidized. The quantity of phenol present must therefore be determined by shaking up with caustic soda. The anthraquinone-test of Meister, Lucius, and Brünig is universally employed for the valuation of anthracene. It was proposed that a special committee should be appointed in order to decide upon the best form of apparatus for estimation by means of fractional distillation.—D. E. J.

#### IV.—COLOURING MATTERS AND DYES, Etc.

***The History of Alizarin Blue.*** A. Scheurer. *Dingl. Polyt. Journ.* 253, 297, 1884.

In September, 1875, a communication was made to the Comité de Chimie, by G. Schaeffer in reference to the change first noticed by Strobel which alizarin red undergoes when subjected to the action of nitrous fumes. Schaeffer found that this change of colour was permanent, and shortly afterwards Rosenstiehl recognised

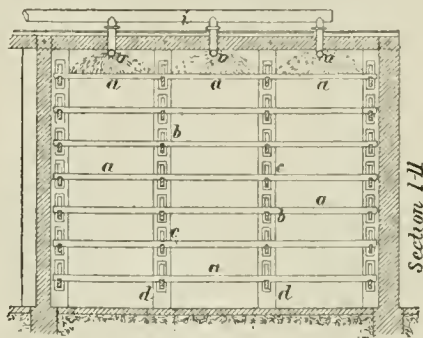
the orange colouring matter so formed as mononitro-alizarin and introduced alizarin orange into commerce. Two years later, in June 1877, Prud'homme published his discovery of two colouring matters, one blue and the other brown, obtained simultaneously by warming a mixture of mononitroalizarin (alizarin orange) with glycerol and sulphuric acid at a temperature of  $200^{\circ}$  C. In a few months these colouring matters were being manufactured, and it became apparent that there was no occasion for the temperature of the mixture to be taken so high as  $200^{\circ}$  C, the formation of the colouring matters taking place even at about  $100^{\circ}$ . The brown colouring matter, Brunck recognised as amidoalizarin, formed as a by-product of the reaction, and obtainable by the reduction of nitroalizarin with tin salt, or grape sugar in presence of sodium hydrate. The blue dye was isolated in the crystalline state by Brunck, and its most important properties described. In December 1877, the Badische Anilin und Soda-Fabrik, introduced alizarin blue into commerce in the form of a paste containing 10 per cent. The practical application of the new product was attended by several difficulties owing to its insolubility in water, acetic acid, and saline solutions. In fact, as Koechlin and Prud'homme pointed out, this colouring matter had properties in common both with indigo and alizarin, being like the former reducible in alkaline solution to a vat, like the latter in uniting with metallic mordants. The tints, however, which were obtained by the vat process lacked solidity. For the production of the purest and fastest shades, Dollfus advised the application of nickel mordants; such have again recently come into favour for alizarin blue steam colours, the tint being a purer green with less tendency to violet than is obtained with the use of chromium acetate. The blue fixed with the aid of chromium acetate, as recommended by Koechlin and Prud'homme, is very slightly affected by the hypochlorites and certain other reagents, but in the light it gradually changes to a neutral grey. Whilst upon the one hand, alizarin blue offers to oxidizing agents a greater resistance than indigo, it is, upon the other hand more readily altered by light than the latter. This behaviour, as Witz considers, is certainly anomalous, and would seem to indicate that the best method for fixing the alizarin blue has yet to be discovered. The most recent advance consists in bringing into commerce alizarin blue in a soluble form. Koechlin first succeeded in fixing coerulein by the aid of alkaline bisulphites, and Prud'homme studied the reaction and found that like an aldehyde or ketone, coerulein could combine with bisulphites and form a colourless crystalline product. In the same manner Brunck has succeeded after considerable difficulty in combining alizarin blue with sodium hydrogen sulphite to form a soluble double salt, which is fixed by the aid of chromium acetate, and yields pure tints which resist the action of light better than indigo. This *alizarin blue S* is a dyestuff of considerable value, and in spite of its high price compares well with alizarin. The composition of alizarin blue has been investigated by Gräbe. Figures were obtained leading to the formula  $C_{17}H_9NO_4$  for alizarin blue and  $C_{17}H_9NO_4 \cdot 2NaHSO_3$  for *alizarin blue S*.—W. D. B.

*Apparatus for the Preparation of White Lead.* Diigl. Polyt. Journ. 253, 296, 1884.

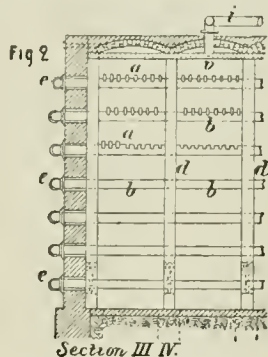
WITH the object of accelerating the formation of the white lead and of lessening the amount of dust which usually escapes upon opening the chambers, H. Kirberg (Ger. Pat. 27398, November 28, 1883), has devised the apparatus here figured. The lead plates are suspended somewhat as in slate roofing, upon the laths *a* which are supported by the carriers *b*. The carriers *b* run from one end of the chamber, through the slits *c* in the upright supports *d* to the other, the end *e* projecting without the chamber wall. As indicated in fig. 4 the carriers *b* are suspended in the slits *c* by the stirrups *f* of brass wire, which turn freely in the direction of the length of the carriers *b* around the cross-pin *g*. The

carriers can thus be moved backwards and forwards by striking the free end *e* with a hammer. The shock is

Fig 1



thus communicated to the laths *a* and therefore to the lead plates suspended thereupon, the pulverulent masses



of white lead formed are thus shaken off and a fresh surface of metal exposed. The perforations in the chamber

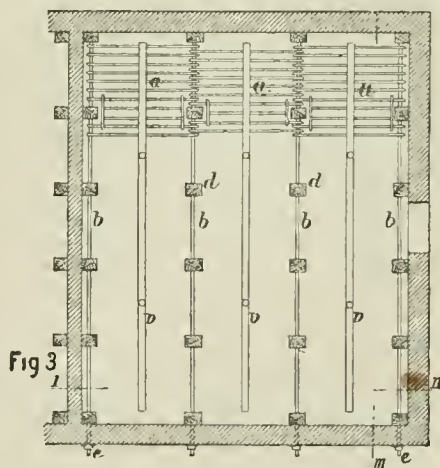
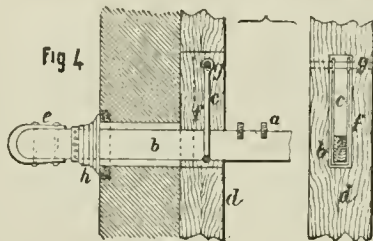


Fig 4



walls through which the carriers *b* pass are kept tight by means of the india-rubber collars *h*. The escape of



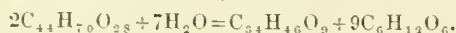
white lead dust upon opening the chambers is prevented by previously spraying the interior with water supplied by the main *v* to the perforated copper tubes *w* which run the whole length of the chamber close to the roof. The masses of white lead upon the lead plates are thus removed.

—W. D. B.

*Substances occurring in Saffron.* R. Kayser. Ber. 17, 2228.

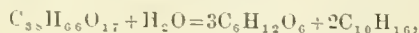
OWING to the methods used for isolating these substances, they have never yet been obtained pure, but have always been accompanied by their decomposition products.

Saffron was distilled with water in a current of carbon dioxide, the distillate extracted with ether, and the ether removed also in a stream of carbon dioxide. The oil thus obtained is colourless, thin, smells of saffron and is a terpene  $C_{10}H_{16}$ . Saffron was extracted with ether, then with water, and the aqueous solution shaken with purified animal charcoal; the charcoal was washed, dried, and extracted with alcohol, when the crocin was obtained pure as a yellow powder. A solution of crocin is not precipitated by lead acetate or lime water, but on heating, it is decomposed into a sugar and crocetin, which gives rise to an orange precipitate. Crocin has the composition  $C_{44}H_{76}O_{28}$ . Crocin boiled with dilute hydrochloric acid in a current of carbon dioxide gives an orange-yellow precipitate of crocetin; no volatile products are formed; while crocin is easily soluble in water, scarcely in alcohol and ether, crocetin is insoluble in water, but easily soluble in alcohol and in ether. Crocetin is  $C_{34}H_{44}O_9$ , and its formation from crocin is expressed:—



The *saffron sugar* or *crocose* is a crystalline body, is dextrorotatory, and is characterised by reducing Fehling's solution only half as strongly as dextrose, viz., 1 gram precipitated 0.877 gram cuprous oxide.

By long continued extraction of dry saffron with ether, crystals gradually separate in the ether flask, which are drained and recrystallized; they are colourless, melt at  $75^\circ$ , and are very bitter. The bitter principle of saffron, or *picrocrocin*, has the composition  $C_{38}H_{66}O_{17}$ , and is a glucoside; its aqueous solution reduces Fehling's solution only on heating, a green precipitate being at the same time formed; heated with dilute acids it becomes turbid, owing to the separation of an oil, which is a terpene and smells of saffron, and is in all probability identical with the oil existing ready formed in the saffron, and the aqueous solution now reduces the copper solution in exactly the same way as crocose. Picrocrocin is thus decomposed in the following manner:



and is an interesting case of a glucoside decomposing into a sugar and a terpene. The saffron was obtained from *Crocus electus* Gatin.—H. B.

#### On Resorcinol.

FROM researches carried out on a large scale at the factory of E. Merck, in Darmstadt, it appears that when resorcinol is fused with caustic soda, and the neutralized fusion extracted with ether, a residue remains, which after recrystallization, contains from 25 to 35 per cent. of diresorcinol. Benedikt (Monatsh. für Chemie 1884, 177) separates diresorcinol from phloroglucol by dissolving in 7-8 parts of boiling water, and allowing to cool to about  $30^\circ$ , when the greater part of the diresorcinol separates in long crystalline needles. These must be rapidly filtered off, when a small further portion will crystallize from the filtrate, the granular crystals of phloroglucol only separating after some standing. A single recrystallization will yield diresorcinol perfectly pure. It melts at  $310^\circ$ . It is much more difficult to free phloroglucol from diresorcinol. It may be obtained approximately pure by fractional crystallization, but traces of diresorcinol are retained. Diresorcinol yields, with acetic anhydride and sodium acetate,  $C_{12}H_8(OC_2H_3O)_4$ , with fuming nitric acid  $C_{12}(NO_2)_6(OH)_4$ , with bromine

$C_{12}Br_6(OBr)_4$ , and with phthalic anhydride and chloride of tin, diresorcinol-phthalein  $C_{12}H_{10}O_4 + C_8H_4O_3 = C_{20}H_{12}O_6 + H_2O$ . —H. R. P.

#### Hæmatoxylin and Brazilin.

ACCORDING to Bucha (Berichte Deutsch. Chem. Ges. 1884, 683), to produce monobromacetyl hæmatoxylin, 5grm. acetyl hæmatoxylin are dissolved in glacial acetic acid, and an equal quantity of bromine also dissolved in glacial acetic acid is added drop by drop. A slight evolution of hydrobromic acid commences, and after standing an hour the bromised compound may be precipitated with aqueous sulphurous acid, filtered off, dried, and recrystallized from alcohol. It forms fine colourless needles, which are soluble in chloroform, glacial acetic acid, and benzene; and after repeated recrystallization, melt at  $210^\circ$ , and have the composition  $C_{16}H_5BrO_6(C_{12}H_9O_5)_2$ . On heating with alkalis, the acetyl-ether is saponified, and bromhæmatoxylin formed, which like hæmatoxylin, is soluble in soda and potash solutions with a blue, and in ammonia with a red colour. If acetyl hæmatoxylin be dissolved in hot acetic acid, and bromine also dissolved in glacial acetic acid be dropped into the boiling liquid, a separation of a brown crystalline body soon commences with free evolution of hydrobromic acid. This body forms a red solution in potash, which rapidly becomes brown.

Acetylbrasilin behaves in most respects similarly to acetyl hæmatoxylin, forming in the cold a monobromotetracetylbrasilin, which is less freely soluble in alcohol than the corresponding hæmatoxylin derivative.

—H. R. P.

*On Condensation Products of Aromatic Bases with aldehydes.* By O. Fischer and C. Schmidt. Ber. Chem. Ges. 1884, p. 1889.

THE bases obtained by the action of dimethyl and diethyl aniline upon ortho-nitro-benzaldehyde and vanillin in the presence of a dehydrating agent (zinc chloride, anhydrous oxalic acid, etc.) form the subject of this investigation.

I. Ortho-nitro-benzaldehyde and dimethylaniline.

The triphenylmethane base is readily obtained from these bodies by the following process:—One part ortho-nitro-benzaldehyde, prepared by Einhorn's method (Ber. Chem. Ges. 1884, p. 119) is heated on a water-bath, with from three to four times its weight of dimethyl aniline, one part of zinc chloride being gradually added. The mixture must be well stirred, and the temperature should not be allowed to rise above  $100^\circ$ , in order to avoid oxidation. A semi-solid, brownish-yellow melt is obtained, from which the excess of dimethyl aniline and zinc chloride are separated by boiling it up with steam. In order to further purify the product, which separates from the zinc chloride solution in the form of a yellow powder, it is extracted by boiling with small quantities of alcohol, and is then recrystallised from a mixture of alcohol and benzene. Thus beautiful golden yellow prisms, melting at  $159-160^\circ$ , are obtained, which unlike most triphenylmethane derivatives contain no benzene of crystallization.

*Colourbase* (Ortho-nitro-malachite green).—To obtain this, the pure crystallized condensation product described above is suspended in water to which three molecules of 50 per cent. sulphuric acid are added. Lead peroxide is then gradually introduced into the solution which must be vigorously agitated. After having been heated on a water-bath for several hours to complete the oxidation, the solution is separated by filtration from the lead sulphate formed. To the filtrate common salt is added, whereby the colouring matter is precipitated. The green precipitate is dissolved in hot water, and the pure colourbase separated from this solution by adding caustic soda and shaking the alkaline solution with ether. From the ethereal solution the base crystallizes in small yellowish-red sparkling crystals, melting at  $163^\circ$ . The neutral salts of this base have an intense green colour with a marked bluish tinge.

*Reduction-Product of the Nitro-Leuco-Base.*—By the addition of zinc dust to an acid solution of the base (ortho-nitro-tetramethyl-diamido-triphenylmethane) an

intense red coloration is produced; which after a time changes into yellow and at last disappears. From this benzene solution the base is separated by the addition of an excess of strong ammonia and dissolving out with benzene. From this solution the benzene-compound separates in colourless crystals melting at  $134-135^{\circ}$ . The behaviour of this base towards oxidizing agents is very characteristic. By means of lead peroxide, or manganese, and a mineral acid, a blue colour is produced, which soon disappears again; in an acetic acid solution an intense blue coloration ensues, which is obtained still more perfect by adding to an alcoholic solution of the base a little acetic acid and then a hot solution of chloranil. By employing arsenic acid as an oxidizing agent a reddish brown colouring matter is produced which shows the characteristic fluorescence of the chrysanilines, and belongs most likely to that class of bodies. By treating the acetyl leuco-base in the usual manner with manganese and a mineral acid, a malachite-green colouring matter is formed. By eliminating the acetyl group, a bluish green colouring matter is obtained, which doubtless represents the normal oxidation product—Ortho-amido-malachite-green.

#### II. Ortho-nitro-benzaldehyde and diethylaniline.

The condensation of these two substances takes place as readily as that of dimethylaniline and ortho-nitro-benzaldehyde, when a less energetic dehydrating agent, preferably anhydrous oxalic acid, is employed. The mode of preparation is the same as in I., only using  $1\frac{1}{2}$  parts of anhydrous oxalic acid instead of one of zinc chloride. The crystals of the base thus obtained resemble in colour those of potassium bichromate and melt at  $109-110^{\circ}$ . By oxidation an intense bluish green is produced. Ortho-amido-tetraethyldiamido-triphenylmethane, which is obtained in the same manner as the methyl-compound, resembles this in its behaviour towards oxidizing agents.

#### III. Vanillin and dimethylaniline.

Vanillin is dissolved in an excess of dimethylaniline; to the solution powdered zinc chloride is gradually added. The mixture is heated for 15 to 20 hours to  $100^{\circ}$ , and then for 2 to 3 hours to  $105-110^{\circ}$ . The product, after being freed from dimethylaniline and zinc chloride is recrystallized from ether. The crystals possess a faint pink colour and melt at  $135-136^{\circ}$ . By oxidation with lead peroxide and sulphuric acid or with chloranil in an alcoholic solution purple colours are produced, which like those obtained from the leuco-base from para-oxy-benzaldehyde and dimethylaniline show a peculiar dichroism.—F. M.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

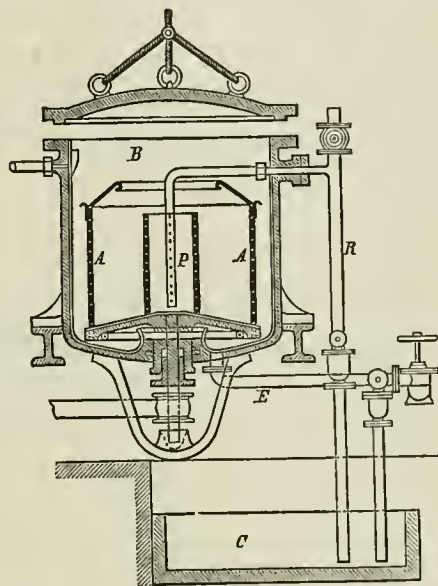
*A New Treatment of Cuscin.* Dingl. Polyt. Journ. 253, 351.

ARMAND DOLLFUS (Moniteur de la Teinture), proposes to treat casein with nitric acid, so as to make a cheap and satisfactory substitute for albumin in calico printing. Ordinary nitric acid quickly converts casein in the cold into nitrocasein, a yellow substance, which must be washed with tepid water till of neutral reaction, and then heated with caustic soda (of which only the necessary quantity should be used), till it is dissolved; enough water being added to reduce it to a suitable consistency for printing. By steaming, it is so firmly fixed in the cloth that it withstands the most vigorous soaping and rubbing, and even chlorine. Indeed alkaline matters and chlorine appear to fix these nitrocasein colours, while they are weakened by acids. The tone of the colour is a yellowish chamois; it may, however, be modified by the addition of ochre, lampblack, madder extract, etc., as required. Employed as a thickening and fixing material it is evident that it will dull bright colours, with the exception of Guignet's green, and red lead, to which it only gives a yellower tint. The dark colours which contain considerable quantities of insoluble matter are less permanent than the lighter ones. Nitrocasein mixes well with egg and blood albumen. If casein be treated with weaker nitric acid, similar changes take place; but if as weak as  $27^{\circ}$  (presumably Beaumé) a white product is obtained, which, though it does not fix

so well by steaming as the yellow, is much better than unaltered casein, and mixes well with albumen, which gives it the necessary solidity.—H. R. P.

*Apparatus for Bleaching or Dyeing Yarns and Goods in Vacuo.* By W. Pornitz. Dingl. Polyt. Journ. 254, 63.

MANY attempts have been made to facilitate the penetration of textile fabrics by the dyeing and bleaching solutions, with which they require to be treated, by carrying out the treatment in vacuo, (i.e., in such apparatus as shall allow of the air being withdrawn. (c.f. Ibid 249, 88.) The apparatus figured in the annexed drawing (Austrian Pat. Jan. 15, 1884) although not



essentially different from those already in use, embodies some important improvements in detail. It consists of a drum A, the sides of which are constructed of stout netting, carried on a vertical axis working through a stuffing box, which is fitted in the bottom of the outer or containing vessel or kier B. The air can be exhausted from B by means of an air-pump. A contains a central division P, also constructed of netting, into which is inserted the extremity of the tube R, after being twice bent at a right angle. P is also in direct connection with the efflux tube E, E and R serving to convey the dye or bleach solutions to and from the reservoir C. The combination of the rotary motion communicated to A, which contains the goods to be dyed or bleached, with the very thorough penetration and circulation of the liquids effected by means of the vacuum established in B, is found to be eminently favourable to the rapidity and evenness of the dye or bleach.—C. F. C.

*Formation of Persulphocyanogen by Electrolysis.* By F. Goppelsroeder. Dingl. Journ. 254, 83.

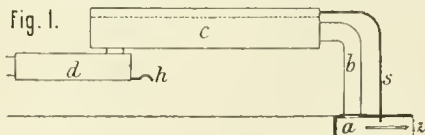
THE author has observed the formation of this compound, to which Gerhardt and Laurent assigned the formula  $C_2N_2HS_2$ , in the electrolysis of solutions of potassium sulphocyanide at the boiling temperature. Its separation at the positive electrode is accompanied by the development of a strongly acid reaction, while at the negative a considerable evolution of gas takes place. The author has succeeded in depositing this colouring matter upon cotton and woollen goods. In a postscript is mentioned the almost simultaneous appearance of a note in the current vol. of the Berl. Ber., p. 252, by A. Lidow, describing similar observations. The author is prosecuting the investigation of the subject.—C. F. C.



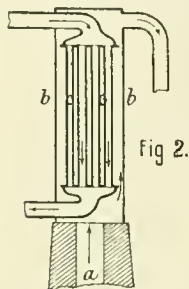
## VII.—ACIDS, ALKALIS, AND SALTS.

*New Apparatus for Obtaining Ammonia.* Dingl. Polyt. Journ. 253, 339, 1884. F. Lorenz, Ger. Pat. No. 26638, 1882.

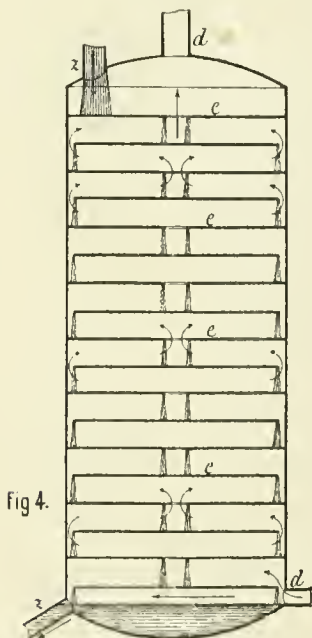
To obtain ammonia and tar from hot gases, conduits them into a passage *a* (fig. 1) in the lowest part of the apparatus, from which they rise by the tube *b*, into a broad and long chamber *c*, of which the cover consists of



a condenser filled with water, from which a stream flows through the tube *s*, on the plate *z*, to saturate the gases with moisture. The cooled gases pass through the canal *d* into an iron tower, through which water trickles, and this, together with the liquid condensed in *d* and *c* is carried off by the tube *h*. The gases are then passed through a second tower supplied with acid.

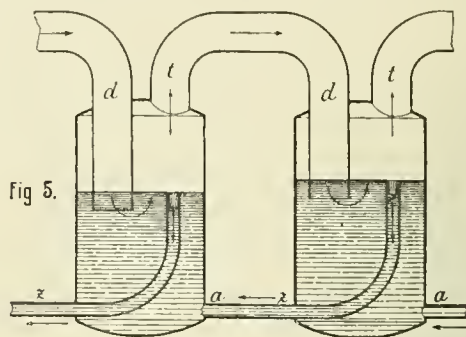


In the supplementary patent, No. 26976, a regenerator, shown in Fig. 2, is inserted in the canal *b*, through which the gases are made to pass between the two towers, in

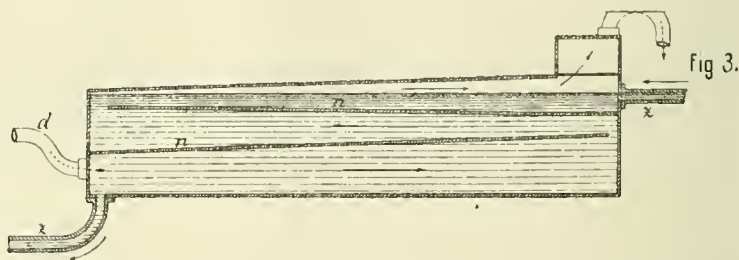


order to be again heated, so that the ammonia may be better absorbed by the sulphuric acid.

J. Young, Ger. Pat. No. 27034, 1883, to obtain ammonia from sewage, and the effluent waters of sugar manufactories, employs a series of long boxes. Fig. 3,



arranged like steps, and provided with inlet and outlet pipes *z*, and a steam pipe *d*, and with false bottoms *m*, inclined in such a way that when the boxes are filled



with the liquid, which is previously warmed and treated with lime, the steam which is admitted at the bottom can be sucked by means of a vacuum back and forwards through the whole battery of boxes, while fresh liquid flows continuously in at the top, and out at the bottom. Instead of the boxes, cylinders may be employed as shown in Fig. 4.

J. Duncan, Ger. Pat. No. 27148, 1883, patents a very similar process, in which cylindrical vessels (Fig. 5) are substituted for the boxes, and so considerable a vacuum employed, that the boiling point never exceeds 30° C.

—H. R. P.

*Sodio-calcium Carbonates.* Formation in the Soda processes. C. Reidemeister. Dingl. Journ. 254, 90, (c.f. 242, 294.)

THE author has observed the formation, in addition to artificial Gay-Lussite ( $\text{Na}_2\text{CO}_3 \cdot \text{CaCO}_3 \cdot 5\text{H}_2\text{O}$ ) of rhombic plates, of the compound  $2\text{Na}_2\text{CO}_3 \cdot \text{CaCO}_3 \cdot 5\text{H}_2\text{O}$ , during the process of concentrating soda liquors. The former salt tends to form in weak liquors at temperatures under 40°, the latter in more concentrated liquors and at temperatures above 40°. (See also this Journal, vol. 1, pages 85 to 87.)—C. F. C.

*The State of the German Alkali Industry and other Manufacturing Processes connected therewith,* by R. Hasenclever. Chem. Ind. 1884, p. 78-86.

#### Sulphuric Acid.

IN the last few years the sulphuric acid industry has largely extended, as will be seen from the following table, showing the production of pyrites in Germany:—

Year.	Siegen.	Geslar.	Rest of Prussia.	Total.
	Tons.	Tons.	Tons.	Tons.
1862	11,850	—	7,461	22,311
1863	28,765	—	5,931	34,699
1864	29,115	—	3,437	32,552
1865	31,060	—	4,187	38,217
1866	50,875	—	4,302	55,177
1867	71,835	1,599	4,756	78,190
1868	90,100	2,635	3,953	96,688
1869	64,789	2,689	6,391	73,872
1870	92,048	3,225	3,191	98,461
1871	110,432	3,321	4,574	118,330
1872	114,745	3,640	961	149,349
1873	123,172	1,217	3,748	128,137
1874	120,326	1,306	5,074	126,796
1875	110,899	904	12,173	123,976
1876	88,397	1,331	21,071	110,802
1877	67,782	375	32,761	100,918
1878	60,960	23	31,767	95,750
1879	60,281	27	40,118	100,426
1880	62,296	25	49,914	112,235
1881	76,437	34	48,451	124,922
1882	111,159	88	46,714	157,961

The acid chiefly sold has a specific gravity 1.711 (142° Tw.) at 50° C. It is still principally made from German pyrites, the lumps being burned in the usual kilns with movable bars, whereas the smalls are roasted in the Maletta or Perret shelf burner. The use of Spanish pyrites increases from year to year. These were first introduced in 1877, and are now employed in fifteen works, which send their burned ore either to Duisburg or to Hamburg for the extraction of copper. From the beginning of next year the price of the sulphur in Spanish pyrites will be reduced by the companies, whereby the consumption of German stone will probably decrease. There are also roasted, small quantities of Hungarian and Norwegian pyrites. Furthermore, considerable quantities of acid are recovered from certain flue gases, and by roasting blende. Only little acid is made from brimstone and spent oxide. Assuming that 100 parts of German pyrites with 42% S yield 140 parts of  $\text{SO}_4\text{H}_2$  of 142° Tw., that 100 parts Spanish pyrites with 48.49% S yield 165 parts of  $\text{SO}_4\text{H}_2$  of 142° Tw., and that 100 parts Hungarian and Norwegian pyrites with 45.46% S yield 155 parts of 142° Tw., the total production of sulphuric acid can be stated as follows:—

From German pyrites, 130,892 tons* a 140 parts....	183,249 tons.
" Spanish " 55,000 " a 165 " "	90,750 "
" Hungarian and Norwegian pyrites, 9,000 tons a 155 parts .....	13,950 "
At Oker, Freiberg, Mansfeld .....	40,200 "
From zinc blende .....	30,000 "

358,149 tons  
at 142° Tw. (60° B)

With regard to the use of blende there exists a prejudice that the gases thus generated are not favourable for the vitriol manufacture, as they are not rich enough in sulphurous acid. By the following calculation the author shows that this view is erroneous. 100kg. sulphur burning to sulphurous acid, require 69.8cbm. oxygen, and yield 69.8cm. sulphurous acid. This quantity of oxygen

is mixed with 262.2cbm. nitrogen. To convert 69.8cbm. sulphurous acid into sulphuric acid, we further require 34.9cbm. oxygen, which are mixed with 131.1cbm. nitrogen. Since the gases should contain six per cent. free oxygen at the "chamber escape," we have to introduce 143.8cbm. air in excess. The gases from 100kg. sulphur will therefore amount to 69.8+262.2+34.9+131.1+143.8=641.8cbm., consisting of

10.64 vol.%  $\text{SO}_2$   
10.36 " O  
79.00 " N

If we perform the same calculation with iron pyrites ( $\text{FeS}_2$ ) and zinc blende ( $\text{ZnS}$ ), we obtain the following result:—

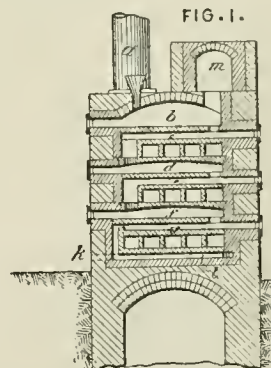
For 100kg.  $\text{FeS}_2$   
37.2 + 52.3+140+18.6+70 + 104.9=233.0cbm.  
Consisting of 8.79 vol.%  $\text{SO}_2$   
9.59 " O  
81.62 " N  
For 100kg.  $\text{ZnS}$   
23 + 68.5+11.5 + 43.2+11.5+43.2+75.8=276.7cbm.  
Consisting of 8.31 vol.%  $\text{SO}_2$   
14.05 " O  
77.64 " N

The gas mixture from 100kg. therefore consists of

	Sulphur.	Pyrites.	Zinc blende.
cbm. $\text{SO}_2$ .....	69.8	37.2	23.0
" N .....	262.2	192.3	129.7
" air .....	309.8	193.5	124.0
	611.8	423.0	276.7

orexpressed in vol.%  $\text{SO}_2$ , 10.6 8.8 8.3  
whereas the total volume of the gases per 100kg. of real sulphur in brimstone, pyrites and zinc blende amounts to 3nlp. Pgrit. zinc bl. cbm. 612, 800, 810.

We therefore find that the percentage of sulphurous acid in the chamber gases from blende is only slightly different from that from pyrites; but on roasting the former the chamber-room must be enlarged in the proportion of 800 to 840. For burning blende Hasenelever and Helbig's furnace is still chiefly employed, but the ore is desulphurized in it only very incompletely. Lately kilns with mechanical agitators have been tried. That of the Vieille Montagne Co., of Oberhausen, consists of three horizontal agitators, turning round a common vertical shaft. At present only one experimental furnace is at work, which is said to give satisfactory results. A firm in Upper Silesia has patented a kiln consisting of three combined shafts, worked with hot air. The "Rhenania" (of Aix-la-Chapelle) made experiments with roasting



blende in a horizontal cast-iron pipe of 40cm. diameter, in which the ore travelled from one end to the other by means of a screw on the now well-known Thelen plan. The pipe was heated from the outside. The blende contained 30% S to begin with, and samples, taken every three hours, tested 23.4, 20.4, 15.2, 10.86, 6.9, 4.4, 4.2% S respectively. On carrying out the experiment on a larger scale, a half-round cast-iron pan (2.5m. diameter and 7.0m. long) was used, but with unsatisfactory results, as the ore could not be protected from cooling down too far. Lastly, the recently designed furnace of Messrs. Eichhorn and Liebig was tried. It consists of six, better of eight horizontal shelves, on which the ore travels from the top to the bottom. The powdered blende is emptied into the

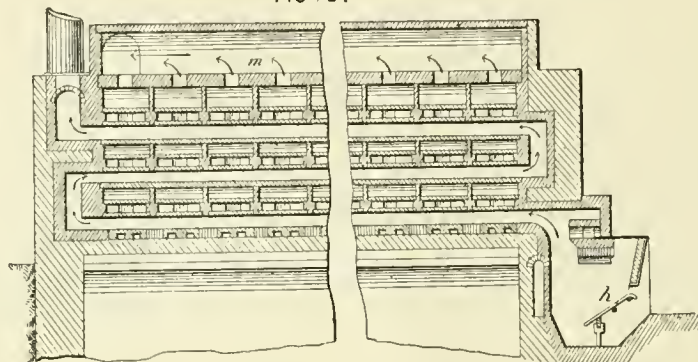
\* The total production of pyrites in 1882, as shown in the table, was 158,409 tons; less export 27,517; leaves German consumption 130,892 tons.



hopper *a* (Figs. 1 and 2), and spread on all shelves as uniformly as possible, in a layer 4cm. high. The fireplace *h* heats the furnace, and the fire-gases pass upwards in the direction of the arrows in the diagram, heating the firebrick shelves and escape at *i* into the chimney. Heating with gas is preferable, since a "plus pressure" can be kept up in the flues, preventing the escape of sulphurous acid into the atmosphere. If the blende on the shelf

lowest one being heated by the fireplace *h*. The ore is emptied into the hopper, and moves downwards from shelf to shelf. The sulphurous acid generated passes into the chambers through *m* in the direction of the arrows. But the blende could be only roasted down to 8%-10% S. It appears that as the fire-gases heated the bottom shelf only, and the distance from one shelf to the other was 30cm., the radiant heat could not act properly.

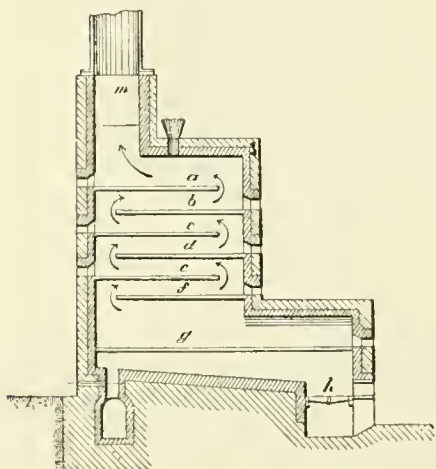
FIG. 2.



*g* is desulphurized, it is withdrawn through a working door, and the charge on the next higher shelf *f* is "shoved down" to *g* and so forth, whereas, fresh material is charged on the top shelf *a*, as soon as it has become empty. The air necessary for roasting enters at *k*, is heated by passing through the flues *l*, and after travelling over the shelves *g*, *f*, *e*, *d*, and so forth, is conveyed as sulphurous acid through *m* to the leaden chambers. Up to the present 3000kg. blende were worked up per 24 hours; every six hours a charge is drawn, and the ore remains in the furnace for 36 hours. On different days the following results were obtained with the same kind of blende:—

	Sample	A	B	C	D
The ore at <i>a</i> contained	S	31.2	31.2	31.2	31.2
" <i>b</i> "	"	28.0	23.8	24.3	21.2
" <i>c</i> "	"	24.8	22.7	19.7	21.5
" <i>d</i> "	"	16.1	16.5	12.3	17.3
" <i>e</i> "	"	8.8	12.5	9.9	2
" <i>f</i> "	"	7.8	7.8	5.4	5.6
" <i>g</i> "	"	0.99	0.90	1.29	1.33

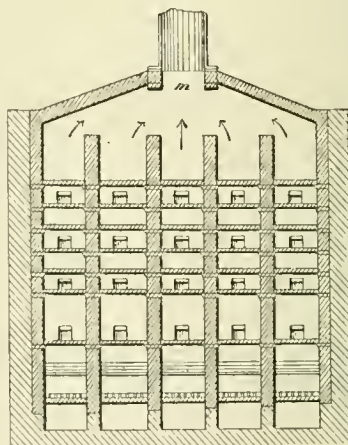
FIG. 3.



Figs. 3 and 4 represent a furnace which was designed and built in 1865 at Stolberg, by Godin, and resembles in the principle that of Eichhorn and Liebig. It also consists of a series of horizontal shelves *a*, *b*, *c*, *d*, *e*, *f*, *g*, the

The inventors, Eichhorn and Liebig, choose 13cm. as a suitable space between two shelves. The "Rhenania" is going to alter all its furnaces on this new plan. As regards the construction of the leaden chambers, no important alterations have been lately made, except that, in order to save lead, the width has been generally increased. Instead of the rectangular cross section, Hasenleiver has cut off the top corners, because this shape conforms not only better to the construction of a roof, but seems also to be more suitable to the passage of the gases. Assuming, according to Schwarzenberg (Bolley, Handbuch d. chem. Technologie, p. 399), that the gases require  $5\frac{1}{2}$  hours from their entering the chambers till their departure, we can suppose that the gases travel with a spiral

FIG. 4.



movement, and do not fill up the whole room uniformly. Such a spiral motion is, however, rather caused by the existence of rectangular corners than if the cross section approaches the shape of a circle. There is also the consideration of a slight saving in lead. Director Stroof, of the Griesheim works, allows the gases to escape from several tunnels in the long side of a chamber. The same works have also taken up again Marignac's experiments for making highly concentrated sulphuric acid and monohydrate by strong cooling and crystallization. For the

carriage of sulphuric acid, iron barrels and bottles have partially replaced the use of glass carboys. Messrs. Vorster and Grüneberg, in order to facilitate the transport of liquid acids, propose to mix them with "Kieselguhr," which absorbs three parts of sulphuric acid, forming a solid mass, which can be used directly for many purposes, or it must be lixiviated if a pure acid is wanted.

**Salt Cake and Hydrochloric Acid Manufacture.**—No improvements have been recently introduced. The Hargreaves process has been unable to make its way into Germany; and Jones' and Mactear's mechanical furnaces have been confined to the manufacture of sulphate of potash. The price of hydrochloric acid has, on the whole, remained unchanged in the last few years, and in many cases it was replaced by oil of vitriol, which has considerably receded in price. Lately, however, a new stimulus has been given to the hydrochloric acid manufacture by the Scheibler process of working up the slags of the Thomas Gilchrist steel process for the extraction of phosphoric acid. The slags have the following composition:—

	A	B	C
P <sub>2</sub> O <sub>5</sub> .....	16.79 .....	17.23 .....	19.20 .....
SiO <sub>2</sub> .....	7.35 .....	6.60 .....	7.20 .....
CaO .....	50.66 .....	53.28 .....	49.00 .....
MgO .....	7.12 .....	7.50 .....	3.75 .....
Fe <sub>2</sub> O <sub>3</sub> .....	3.98 .....	4.40 .....	1.83 .....
FeO .....	7.85 .....	10.66 .....	9.00 .....
MnO .....	1.71 .....	3.10 .....	4.62 .....
CaS .....	1.06 .....	1.06 .....	0.92 .....

The details of the process have been already published in the Journal. The German hydrochloric acid production in 1882 amounted to

126,150 tons from 18 soda works,	
and 22,000 .....	8 potash ..
altogether 148,150 tons of 31½° Tw. (20° B.)	

**Soda Ash, &c.**—The manufacture of soda ash has risen considerably in the last few years. The total production of calcined, caustic and crystallized soda—calculated as 100% Na<sub>2</sub>CO<sub>3</sub>—which in 1877 amounted to 42,500 tons, has increased to 115,500 tons in 1883, of which 56,200 tons are made by the Leblanc, and 59,300 tons by the ammonia process. The price of soda ash, which before 1877 was 23 marks (or shillings) per 100kg. is at present 12 to 14 marks. The price has therefore fallen 45%, whereas the production has more than doubled. As only very small quantities of soda are exported, it must be concluded that the consumption of this article has extended immensely since 1877. Hasenclever ascribes this happy result to the change of the German commercial policy in 1879 from free trade to protection, and argues that, although the alkali is now produced at home, the consumer pays for it only half as much as, years ago. The advance of the ammonia-soda process is as victorious in Germany as it is in France and England. The Solvay Co. is steadily enlarging its works, and before long the import of soda into Germany will altogether cease, and will be followed by an overproduction, which must be naturally accompanied by a struggle between the Leblanc-soda and ammonia-soda works. Whereas, in England and in France a *modus vivendi* has been established for the present, the Leblanc soda manufacturers of Germany may prepare themselves for a severe crisis in the near future. Mr. Hasenclever then discusses the results which Mr. Chance, of Birmingham, has obtained in working the Schaffner and Helbig sulphur recovery process. These, as well as Mr. L. Mond's patent for the manufacture of hydrochloric acid as a by-product of the ammonia soda process have been already published in the Journal. In the Solvay process there have been introduced important improvements, which resulted in increasing the productive power of each set of apparatus, termed an "element" by M. Solvay. Whereas, an element used to produce 10 tons sodaash per 24 hours, it now yields from 15 to 22, and these last few months even 30 tons. That such improvements mean a smaller loss of ammonia per ton of soda, a

saving in wages and capital outlay for plant, besides other advantages, need scarcely be pointed out. In the Leblanc process the introduction of the Thelen apparatus for boiling down and calcining, a better market for hydrochloric acid, a saving in fuel, and a cheaper price for pyrites have also reduced the costs of production. The author thinks it improbable that many works will discontinue their operations, if a crisis sets in, owing to overproduction. Most of the soda works are owned by limited companies, who go on working for a long time, even without profit. "Reduction of share capital," sighs Mr. Hasenclever, "issue of debenture bonds, and loans with bankers, are among the means in Germany to carry on manufacturing establishments without profit for a very long time." Therefore there need be no fear at present that one process will drive out the other; but it may be safely assumed that German soda works must be satisfied with a small rate of profit for a long time to come.—S. H.

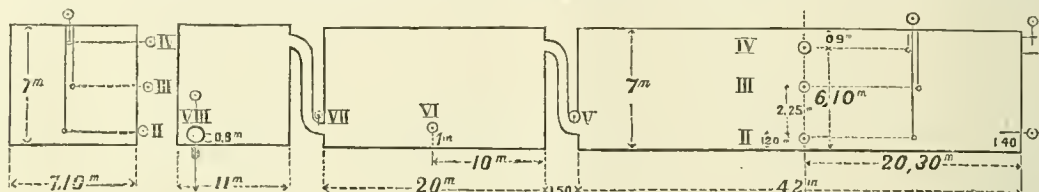
*The Processes of the Sulphuric Acid Chambers.* By G. Lunge, and P. Naef. Chem. Ind., 1884, p. 5.

THE experiments recorded in this paper were chiefly made at the works of Schnorf Brothers at Uetikon near Zürich, where, for several months, a series of chambers with some of the latest improvements was devoted entirely to the purposes of research. Other experiments were also conducted in the British Alkali Works at Widnes. The chamber system at Uetikon had a total cubical contents of 3,650 cbm., and comprised three chambers, No. 1, 42m. long, 7.1m. wide, 7m. high; No. 2, 20×7.1×7m.; No. 3, 11×7.1×7m. The pyrites burner was of the Malétra construction and had 10 compartments. The Glover tower was 3.3m. in diameter, and 9m. high; the Gay-Lussac tower was of unusually large dimensions, viz., 2.4m. in diameter, 17m. high, the cubical contents being 76.8 cbm., that is, 2.1 per cent. of the total chamber space. The system worked under ordinary circumstances in a normal manner; the consumption of nitric acid, of 36° B. was 1.4 per cent. calculated upon the pyrites, or of NaNO<sub>3</sub>, 2.2 per cent. calculated upon the sulphur. In different parts of the system, leaden tubes were soldered in the chamber walls. These tubes held doubly perforated caoutchouc stoppers, through the one perforation a glass tube reaching for 1.5m. in the interior of the chamber passed, in the other a thermometer was fitted. In the places indicated by the Roman numbers in the diagram here given, the leaden tubes were inserted. I., in the connection between the Glover tower and the first chamber; II., III., IV., in the side wall of the first chamber, 20m. from the end, placed vertically above each other at distances of 1.2, 3.5 and 6.1m. respectively from the floor. XII., in the end wall of the first chamber, 1.4m. from the floor; X., in the centre of the cover and in the same vertical plane as II., III., IV.; XI., likewise in the cover, halfway between X. and the end of the chamber; V., in the tube connecting the first and second chambers; VI., in the middle of the side wall of the second chamber, and 1m. from the bottom; VII., in the tube connecting the second and third chambers; VIII., in the tube connecting the third chamber with the Gay-Lussac tower; IX., in the exit tube of the Gay-Lussac tower, opening directly to the air. As regards the analysis of the chamber gases, it was considered desirable, when possible, to estimate N<sub>2</sub>O<sub>3</sub>, N<sub>2</sub>O<sub>4</sub>, NO, SO<sub>2</sub>, O and N, disregarding N<sub>2</sub>O, which could not well be present in more than traces, as the separation of the other nitrogen oxides in presence of a great excess of SO<sub>2</sub> already presented considerable difficulty. For N<sub>2</sub>O<sub>3</sub> and N<sub>2</sub>O<sub>4</sub>, concentrated sulphuric acid had been found by one of the authors, a good absorbent, and by estimating upon the one hand, the total nitrogen in the solution, upon the other, the amount of oxygen absorbed from permanganate to oxidize the N<sub>2</sub>O<sub>3</sub> and N<sub>2</sub>O<sub>4</sub> to N<sub>2</sub>O<sub>5</sub>, an accurate estimation of the N<sub>2</sub>O<sub>3</sub> and N<sub>2</sub>O<sub>4</sub> could be obtained. The method is unfortunately worthless in



presence of  $\text{SO}_2$ . The following method was then had recourse to: The gases freed from  $\text{H}_2\text{SO}_4$  by passing through a long tube filled with cotton-wool, were conducted by means of an aspirator through soda-lye which absorbed  $\text{N}_2\text{O}_3$ ,  $\text{N}_2\text{O}_4$  and  $\text{SO}_2$ , then through permanganate solution for the absorption of NO, the residual gas, consisting of O and N, being collected in the aspirator, and the first estimated directly, the second inferred from the difference. In the soda solution, estimations were made of, (a) the sulphur by oxidation with bromine water and precipitation with barium chloride; (b) the oxygen consumed from permanganate; (c) the nitrogen either in the nitrometer or by the iron method. This plan, for several reasons, had to be abandoned. Experiments with samples of gas, of known composition, showed that sodium hydrate solution brings about the decomposition of nitrous acid with formation of NO, that sulphurous acid expels nitrous acid from its compound with soda, and that sulphurous acid vitiates estimations made by means of the nitrometer; and lastly, owing to the great dilution of the chamber gases with air, a considerable portion of the  $\text{SO}_2$  undergoes oxidation, and estimation (b) becomes of

of each system of tubes, an aspirator consisting of a glass bottle of about 18 litres capacity, and graduated in litres, was attached. The water which flowed from the aspirators was collected and measured at the end of each experiment. The amount of gas drawn through each system of U-tubes was thus arrived at. The unabsorbed portion of the gas which was collected in the aspirator consisted of oxygen and nitrogen. The first was estimated endiometrically by means of pyrogallie acid, or, latterly, in Orsat's apparatus by means of phosphorus, the second was inferred from the difference. The sodium hydrate solution in the U-tubes was treated with bromine water for the oxidation of  $\text{SO}_2$ , acidified with hydrochloric acid and precipitated with barium chloride. To the solution of potassium permanganate, a measured quantity of standard ferrous sulphate was added in excess, the amount in excess being determined by titrating back with  $\frac{\text{N}}{10}$  potassium permanganate solution. As NO could not very well be present, especially when dealing with strongly yellow gases, the small quantity of oxygen absorbed from the permanganate was always calculated into  $\text{N}_2\text{O}_3$ , i.e. of  $\frac{\text{N}}{10}$   $\text{KMnO}_4$  corresponding to 0.558e.c. of  $\text{N}_2\text{O}_3$ . In the sulphuric acid,  $\text{N}_2\text{O}_3$  and  $\text{N}_2\text{O}_4$  were



no value. The final result of many experiments was to prove that soda-lye can only be employed for the absorption of  $\text{N}_2\text{O}_3$  and  $\text{N}_2\text{O}_4$  in cases where it is not desired to separate those two compounds quantitatively. This remark applies specially to the analysis of the gases of the first chamber; on account of the enormous excess of  $\text{SO}_2$  small errors in the determination of that constituent would lead to very large ones in that of the  $\text{N}_2\text{O}_3$  and  $\text{N}_2\text{O}_4$ . No attempt was therefore made to determine the  $\text{N}_2\text{O}_3$  and  $\text{N}_2\text{O}_4$  separately in the gases of the first chamber. The method about to be described was employed for the analysis of the gases of the second and third chambers, and Gay-Lussac tower. The sampling tube fitted as above mentioned in the chamber wall was connected at its outer end with a T-piece, both limbs of which were joined to systems of U-tubes. The first limb led to four U-tubes, three of which were filled with concentrated sulphuric acid, the fourth with a known quantity of  $\frac{\text{N}}{10}$  solution of potassium permanganate. The second limb had continuation in a tube filled with cotton wool for a length of 0.5m., by which means finely divided sulphuric acid was retained. The tube filled with cotton wool was connected with two U-tubes containing soda-lye for the absorption of  $\text{SO}_2$ . At the end

determined by Lunge's method, (Ber. d. Deutsch. Chem. Ges., Vol. 2, p. 1,230), according to which the total nitrogen is estimated by the nitrometer and the oxygen required to form  $\text{N}_2\text{O}_3$ , by titration with  $\frac{\text{N}}{10}$   $\text{KMnO}_4$ . A deduction has, of course, to be made for the oxygen required by the  $\text{SO}_2$ . The researches undertaken are described in detail under different headings.

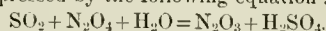
A.—*The absence of nitrogen tetroxide in a chamber system working normally.*—All the experiments in connection with this question were made when the chamber system was working in a normal manner, the gases of the first chamber having a light, of the second a deeper, of the third a dark yellow colour. At the Gay-Lussac tower, slightly yellow fumes were visible. Lasne and Benker's apparatus, though fitted at the works at Uetikon, was not brought into action in any portion of this research. The collection and analysis of the gases of different chambers were made at the same time in the case of the experiments having the same number. In the following table, the amount of the constituents is expressed in volumes per cent. The Roman numerals refer to the position of the sampling tubes as indicated in the diagram given above:—

No. of Expt.	No. of Chamber.	Position of Sampling Tube.	Composition of Gas.				
			O	N	$\text{SO}_2$	$\text{N}_2\text{O}_3$	$\text{N}_2\text{O}_4$
1	3	VIII.	5.86	93.95	0.003	0.184	—
2	3	VIII.	5.71	94.07	0.003	0.209	0.009
	2	VII.	5.82	94.00	0.010	0.166	—
3	3	VIII.	6.99	92.83	0.006	0.173	—
	2	VII.	7.12	92.62	0.127	0.135	—
4	3	VIII.	6.20	93.59	0.0006	0.210	—
	Gay-Lussac Tower.		IX.	—	—	0.029	—
5	3	VIII.	5.89	93.86	0.002	0.230	—
6	3	VIII.	6.00	93.80	0.032	0.173	—

The most important deduction which may be drawn from these figures is that in a chamber system working normally no nitrogen tetroxide is present, the predominating oxide of nitrogen being  $N_2O_3$ , which may be locally reduced by  $SO_2$  to  $NO$ , the  $NO$ , however, being at once oxidized back to  $N_2O_3$ . The same theory as to the function of the  $N_2O_3$  was advanced by Berzelius, and subsequently by Weber, but there was no direct evidence to prove the absence of  $N_2O_4$ . The authors consider that the theory advanced and maintained by one of them for several years, as to the important function discharged by  $N_2O_3$ , has now received by these experiments on the large scale, entire confirmation. As mentioned above, it was not found possible to separate analytically  $N_2O_3$  and  $N_2O_4$  in the gases of the first chamber, the theory is therefore constructed upon the analyses of the gases of the other chambers. But consideration of the following facts will lead to the conclusion that  $N_2O_4$  is likewise absent from the first chamber. It is shown further on that under no circumstances was  $N_2O_4$  found in the second chamber, even when owing to abnormal working there was much of that gas in the third chamber; second, the oxides of nitrogen contain less oxygen in the fore part of the system, where the  $SO_2$  exerts the most energetic reducing action, than in the hinder part; and, as is shown by experiments 22 to 24, the excess of  $NO$  found in the fore part of the first chamber decreases gradually until the other end of that chamber is arrived at. In the above experiments, the amount of oxygen present was found in the majority of cases to be a normal one. In experiment 3, seven per cent. of oxygen was found but no  $N_2O_4$ . From this it would appear that the presence of an excessive quantity of oxygen cannot of itself cause the formation of  $N_2O_4$ . Further confirmation of this is to be obtained from the following research as to the processes which take place under abnormal conditions of work.

B.—*Conditions under which a Formation of Tetroxide of Nitrogen Takes Place.*—Experiments were made to determine whether a formation of  $N_2O_4$  takes place when the amount of nitrogen oxides introduced is such that, viewed through the panes, the chamber gases have a dark yellow colour, and the exit gases from the Gay-Lussac Tower are likewise strongly coloured. The following analytical results show that under such circumstances a formation of  $N_2O_4$  continually takes place:—

in the second chamber only  $N_2O_3$  was present. If, under circumstances the most favourable for its formation,  $N_2O_4$  is found only in the hinder part of the system, and not at all in the first chamber, where the greatest portion of the sulphuric acid is produced, how much less may it be expected to occur in the first chamber when the last contains no oxide of nitrogen but  $N_2O_3$ . The formation of  $N_2O_4$  must be considered as due to a secondary reaction taking place in the hindmost portion of the chamber system under certain conditions, and entirely unconnected with the process proper of sulphuric acid production. The occurrence of  $N_2O_4$  when the chamber gases have a strongly yellow colour, that is to say when a large excess of nitrogen oxides is present, may be ascribed to two causes: first, under such circumstances, the formation of sulphuric acid is so far completed in the first chamber that the amount of sulphuric acid in the last chamber is not sufficient to prevent the oxidation of the nitrogen above the compound  $N_2O_3$  as it is able to when the working is normal. In addition to this it may be pointed out as the second cause that when the chamber gases are very yellow  $SO_2$  does not occur in the last chamber, as it is found to do when the working is normal, exerting a reducing action upon the  $N_2O_4$  which may be expressed by the following equation:—



$N_2O_4$  cannot exist until the  $SO_2$  has been reduced to its minimum proportion, and in chamber gases containing  $N_2O_4$  only very small amounts of  $SO_2$ —from '0004 to '0002 per cent.—are found. The amount of free oxygen does not appear to have any influence upon the formation of  $N_2O_4$ . With a large excess of nitrogen oxides and an abnormally low percentage of oxygen  $N_2O_4$  was found to occur in the third chamber, as the following two analyses show:

## EXPERIMENT 12.

Chamber 3, Sampling tube VIII.		Composition of Gas.
O	.....	4.21 per cent.
N	.....	95.51 "
$N_2O_3$	.....	0.168 "
$N_2O_4$	.....	0.110 "

## EXPERIMENT 13.

Chamber 3, Sampling tube VIII.		Composition of Gas.
O	.....	4.31 per cent.
N	.....	95.46 "
$N_2O_3$	.....	0.170 "
$N_2O_4$	.....	0.118 "

No. of Expt.	No. of Chamber.	Position of Sampling Tube.	Composition of Gas.				
			O	N	$SO_2$	$N_2O_3$	$N_2O_4$
7	3	VIII.	7.13	92.57	0.002	0.182	0.121
		IX.	—	—	—	0.0008	0.083
8	3	VIII.	5.32	91.43	0.0001	0.195	0.051
		VII.	5.61	91.11	0.064	0.189	—
9	2	IX.	—	—	—	0.022	0.002
		VIII.	5.50	91.25	—	0.216	0.005
10	3	IX.	—	—	—	0.018	0.001
		VIII.	5.79	93.95	—	0.213	0.025
11	3	VIII.	5.80	93.91	0.001	0.177	0.111
		VII.	6.01	93.75	0.007	0.231	—

These results derived confirmation from further experiments made at Widnes. Special consideration may be given to experiments 8 and 11. In both cases the third chamber contained a considerable amount of  $N_2O_4$ , whilst

On the other hand the following experiments show that when the amount of nitrogen oxides is normal, even a large excess of free oxygen does not give rise to the formation of  $N_2O_4$ .



No. of Expt.	No. of Chamber.	Position of Sampling Tube.	Composition of Gas.			
			O	N	N <sub>2</sub> O <sub>3</sub>	N <sub>2</sub> O <sub>4</sub>
14	3	VIII.	8.18	91.61	0.180	—
15	3	VIII.	8.59	91.31	0.105	—
	2	VII.	8.67	91.21	0.119	—
16	3	VIII.	9.11	90.77	0.123	—
	2	VII.	9.19	90.68	0.130	—

## II. The Loss of Nitre by the Gay-Lussac Tower.—

From a theoretical point of view the process of sulphuric acid production is a continuous one. Practically, however, there is invariably a loss of nitre, amounting under favorable circumstances to about 2.3 parts of NaNO<sub>3</sub> per 100 of sulphur. This loss is due either to the occurrence of chemical reactions in which the oxides of nitrogen are reduced to N or NO, or to the mechanical removal of nitrogen compounds from the system either in the exit gases, the sulphuric acid, or by accident. The loss, by reason of chemical reactions, has already been studied by one of the authors. The most important results arrived at by this earlier work are that in the Glover tower, there is no appreciable loss by reason of reduction to N or NO, and even in the chambers such a reduction can seldom be detected, and then only locally, near the steam inlet. When the system is working normally the loss of nitre is a mechanical one for by far the greater part, and is to be ascribed mainly to an incomplete absorption in the Gay-Lussac tower, and in a less degree to the removal of acid from the chambers. When largely diluted with air, nitrous fumes can no longer be distinguished by their yellow colour, but when the chamber gases are strongly yellow, yellow fumes are frequently observed at the exit of the Gay-Lussac tower. This loss is ascribed by Lasse and Benker to the small absorptive power for N<sub>2</sub>O<sub>4</sub> possessed by sulphuric acid, hence their process for the reduction of N<sub>2</sub>O<sub>4</sub> to N<sub>2</sub>O<sub>3</sub> by the introduction of SO<sub>2</sub> into the tower. It is very possible that by such procedure the consumption of nitre, calculated upon the sulphuric acid produced, is reduced by reason of the reactions being continued even in the tower, but it has been shown by one of the authors (Lunge, Ber. d. Deutsch. Chem. Ges. 1882, p. 488.) that N<sub>2</sub>O<sub>4</sub> is easily and completely absorbed by sulphuric acid of 60° B., with formation of nitroso-sulphonic acid and nitric acid. The following series of experiments, in which the chamber gases were maintained of a strongly yellow colour, and in which, therefore, the amount of nitrogen oxides in some cases was above the normal, was made with a view of determining whether N<sub>2</sub>O<sub>4</sub> is absorbed with difficulty. Some of the experiments have been recorded above, but are again set down here for purposes of comparison.

### EXPERIMENT 4.

Chamber 3 (VIII).	Gay-Luss.
O .....	6.20
N .....	93.59
SO <sub>2</sub> .....	0.006
N <sub>2</sub> O <sub>3</sub> .....	0.210
N <sub>2</sub> O <sub>4</sub> .....	—

### EXPERIMENT 7.

Chamber 3.	Gay-Luss.	Nitr. Vit. (60° B)
O .....	7.13	—
N .....	92.57	—
SO <sub>2</sub> .....	0.002	—
N <sub>2</sub> O <sub>3</sub> .....	0.182	0.0008
N <sub>2</sub> O <sub>4</sub> .....	0.121	0.083

### EXPERIMENT 8.

Chamber 3.	Chamber 2.	Gay-Luss.	Nitr Vit (60° B)
O .....	5.32	5.61	—
N .....	94.43	94.14	—
SO <sub>2</sub> .....	0.0004	0.061	—
N <sub>2</sub> O <sub>3</sub> .....	0.195	0.189	0.022
N <sub>2</sub> O <sub>4</sub> .....	0.051	—	0.002

### EXPERIMENT 12.

Chamber 3.	Gay-Luss.	Nitr Vit (57° B)
O .....	4.21	—
N .....	95.51	—
N <sub>2</sub> O <sub>3</sub> .....	0.168	0.023
N <sub>2</sub> O <sub>4</sub> .....	0.110	0.053

### EXPERIMENT 9.

Chamber 3.	Gay-Luss.	Nitr Vit (59° B)
O .....	5.50	—
N .....	94.25	—
N <sub>2</sub> O <sub>3</sub> .....	0.246	0.018
N <sub>2</sub> O <sub>4</sub> .....	0.005	0.001

### EXPERIMENT 17.

Chamber 3.	Gay-Luss.	Nitr Nit (59° B)
O .....	5.29	—
N .....	94.47	—
N <sub>2</sub> O <sub>3</sub> .....	0.242	0.015
N <sub>2</sub> O <sub>4</sub> .....	0.0004	—

The absolute loss of nitre in the above experiments may be arrived at if the total volume of gas which leaves the Gay-Lussac tower in a given period be known. As it is not possible to measure this volume with sufficient accuracy, recourse must be had to a process of calculation from the quantity of pyrites burnt, and the proportion of oxygen in the exit gases. (cf. Lunge's Soda Industrie I, p. 209). For the oxidation of 2mols. FeS<sub>2</sub> into Fe<sub>2</sub>O<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>, 15at. of O are required, or 1,300 litres of oxygen, and 4,900 litres of nitrogen for every kilogram of sulphur oxidized. The exit gases contain upon an average 6 vols. p.c. of free oxygen, therefore for 1kilo. of sulphur oxidized 2,100 litres of air in addition to the above quantities are required, and the total volume of gas which for every one kilogram of sulphur leaves the Gay-Lussac tower will be 4,900 + 2,100 = 7,000 litres. In the chamber system at Uetikon 2,700 kilograms S (from 6,000 kilograms of pyrites) were burnt daily; in 24 hours the volume of gas passing from the Gay-Lussac tower was therefore 7 × 2,700 = 18,900 cubic metres. The daily loss of nitre by the non-absorption of N<sub>2</sub>O<sub>3</sub> and N<sub>2</sub>O<sub>4</sub> in the Gay-Lussac tower in experiments 4, 8, 9, made under conditions nearer to the normal than the others is calculated to have been as follows:

No. of Expt.	N <sub>2</sub> O <sub>3</sub>	NaNO <sub>3</sub>			Nitric Acid 36° B		
		Absolute logs in Kilogr.	p.c. calc. on the S	p.c. calc. on the Pyrites	Kilogr.	p.c. calc. on the S	p.c. calc. on the Pyrites
	litres.						
4	5481	41.65	1.54	0.69	61.93	2.29	1.03
8	4317	33.13	1.22	0.55	49.12	1.81	0.81
9	3591	27.29	1.01	0.45	40.57	1.50	0.67

Under ordinary commercial working, the yearly average of loss in the same system was

NaNO <sub>3</sub>		Nitric acid 38° B	
p.c. calc. on the S	p.c. calc. on the Pyrites	p.c. calc. on the S	p.c. calc. on the Pyrites
2.2	1.0	3.1	1.4

Thus in spite of the large dimensions of the Gay-Lussac tower, there was a loss of nitre due to imperfect absorption. In experiment 4, 70 per cent. of the total loss of nitre is to be ascribed to imperfect absorption, in experiment 8, 55 per cent.; in experiment 9, 45.9 per cent. The balance of loss is no doubt partly due to a removal of nitrogen oxides in the chamber acid, but whether the remaining fraction of loss is on account of reduction to NO or N cannot be certainly said. It must be pointed out that the yearly average of the total loss of nitre would most probably be greater than the loss observed in the experiments referred to; hence it is possible that more extended experiments would prove, practically the whole loss to be due to imperfect absorption in the Gay-Lussac tower, when the system was working normally. The authors consider that their results go far to prove that the chemical loss of nitre by reason of a too strong reduction, if not *nil* is too small to be taken into any account. Experiments 4, 8, 9 and 17 show, that, contrary to the usual assumption, *the yellow fumes of the Gay-Lussac exit gas do not consist of N<sub>2</sub>O<sub>4</sub>*. When the colour is only feebly yellow, N<sub>2</sub>O<sub>3</sub> alone is present, but when the chamber gases have a strongly yellow colour, N<sub>2</sub>O<sub>4</sub> is found both in the inlet and outlet pipes of the Gay-Lussac tower. As regards the strength of the acid employed in the Gay-Lussac tower, a number of experiments were made with a view of determining whether an acid stronger than that of 59° B. usually at work would be a more perfect absorbent, and whether any difference could be observed as to the relative absorption of N<sub>2</sub>O<sub>3</sub> and N<sub>2</sub>O<sub>4</sub> by acids of different strengths. It was found that there was no distinct difference between the action of acids of respectively 59° and 60° B, however, acid of 62° B. gave somewhat better results. Experiments upon a small scale showed that by increasing the flow of acid an improved absorption resulted; upon the large scale however, no advantage was obtained by increasing the amount of acid which passed through the tower, and it was also seen that the work which a tower could perform was the same whether it gave a nitrous vitriol containing 1.5 per cent. or only 1.0 per cent. of N<sub>2</sub>O<sub>3</sub>. From the analyses of nitrous vitriol which are given above, it appears that N<sub>2</sub>O<sub>3</sub> is the only oxide of nitrogen present, even in cases where the chamber gases contained N<sub>2</sub>O<sub>4</sub> as well as N<sub>2</sub>O<sub>3</sub>. This fact had previously been pointed out by Lunge (Dingl. Polyt. Journ. 225, 291), and has received confirmation at the hands of Benker and Lasne, and Marsh. It is suggested that statements made by other observers as to the presence of N<sub>2</sub>O<sub>4</sub> were based upon results obtained by unreliable methods of analysis. The authors do not absolutely deny the possibility of N<sub>2</sub>O<sub>4</sub> or HNO<sub>3</sub> occurring in nitrous vitriol but consider their presence extremely improbable when the chambers are working normally. The apparent non-absorption of N<sub>2</sub>O<sub>4</sub> in the Gay-Lussac tower is all the more strange when regard is had to the ease with which sulphuric acid of 60° B. can absorb that gas even when considerably diluted, but whether this anomaly is to be ascribed to the enormous dilution of the gases of the Gay-Lussac tower or to a reduction to N<sub>2</sub>O<sub>3</sub> of the NO<sub>2</sub>H formed by the reaction of N<sub>2</sub>O<sub>4</sub> with H<sub>2</sub>SO<sub>4</sub>, by traces of SO<sub>2</sub> or by the coke of the tower, there is insufficient experimental data to determine. In the experiments given above in this division of the paper, the chamber gases were strongly yellow. In the following series, the chambers were worked light and with distinct excess of SO<sub>2</sub>.

No. of Expt.	Chamber 3 or Gay-Lussac.	Composition of Gas.					
		O	N	N <sub>2</sub> O <sub>3</sub>	N <sub>2</sub> O <sub>4</sub>	NO	SO <sub>2</sub>
18.	Chamber 3 ..	5.81	94.00	0.155	0	0.003	0.037
	Gay-Lussac ..	—	—	trace	trace	0.0046	0.011
19.	Chamber 3 ..	5.79	94.05	0.132	0	0.012	0.013
	Gay-Lussac ..	—	—	0.0001	0	0.0101	0.026
20.	Chamber 3 ..	5.91	93.91	0.111	0	0.020	0.020
	Gay-Lussac ..	—	—	0.0011	0	0.017	0.037
21.	Chamber 3 ..	6.00	93.78	0.169	0.001	0.014	0.036
	Gay-Lussac ..	—	—	0.009	0	0.0066	0.0316

The absolute loss of nitre was found by calculation to be as follows:—

No. of Expt.	N <sub>2</sub> O <sub>3</sub> equivalent to NaNO <sub>3</sub> .			
	Litres per Diem.	Kilos. per Diem.	p.c. calc. on the S.	p.c. calc. on Pyrites.
18	435	3.30	0.13	0.05
19	983	7.47	0.28	0.12
20	1814	13.78	0.51	0.23
21	2324	17.66	0.65	0.29

When the Gay-Lussac exit gases contain SO<sub>2</sub> the amounts of N<sub>2</sub>O<sub>3</sub> and N<sub>2</sub>O<sub>4</sub> are extremely small, but NO occurs, and even in larger quantity than is shown by the analysis on account of the difficulty of absorbing that gas when so much diluted. The loss of nitre, working with light chambers and a small excess of SO<sub>2</sub>, is less than when the chambers are strongly yellow, in the first case the loss is 0.13-0.65 NaNO<sub>3</sub> per 100 S, in the second case 1.0-1.5 NaNO<sub>3</sub> per 100 S. It may, therefore, be found advantageous to work with a very slight excess of SO<sub>2</sub>, or, employing Lasne and Benker's process, to work with yellow chambers and then correct the Gay-Lussac gases with SO<sub>2</sub>. A series of experiments made at the British Alkali Works, Widnes, gave results according very well with those obtained at Uetikon. The loss of nitre was greater at Widnes than at Uetikon, both at the Gay-Lussac tower and throughout the whole system, but the proportion of loss occurring at the Gay-Lussac tower was less at Widnes than at Uetikon.

III. *On the Distribution of the Gases and Progression of the Reactions in the Chambers.*—In these experiments samples of the gases were collected simultaneously at different points of the system. The analyses lead to the following conclusions. With normal working the proportion of SO<sub>2</sub> decreases very rapidly between its point of entry into and the centre of the first chamber, namely from about 7 p.c. to from 1.7 to 1.9 p.c.; 70 p.c. of the SO<sub>2</sub> is thus converted into SO<sub>3</sub> by the time the centre of the first chamber is reached. This agrees well with Hurter's theory. From the centre to the end of the first chamber the vigour of the reaction is much reduced, and only about 4 p.c. of the original SO<sub>2</sub> introduced are here oxidized. Between the entry into and centre of the second chamber about 20 p.c. more of the SO<sub>2</sub> are converted into SO<sub>3</sub>, the gases at the centre were found to contain only 0.2 to 0.4 p.c. of SO<sub>2</sub>. The passage of the gases through the pipe connecting the first and second chambers, thus appears to reinvigorate the reactions. From the centre of the second chamber to the end of the system the oxidation of SO<sub>2</sub> proceeds very slowly. With a too small supply of nitrous fumes, the oxidation of SO<sub>2</sub> in the first chamber is not so rapid as when working normally, the reactions taking place to a greater degree in the second and third chambers. In the middle of the first chamber 3.0 p.c. of SO<sub>2</sub> were found, 59 p.c. of the

\* Note by abstractor, apparently meant for 0.132.



original  $\text{SO}_2$  were thus oxidized between the entry into and middle of that chamber; between the middle and the end of the first chamber 14 p.c. more  $\text{SO}_2$  were oxidized, whilst in the first half of the second chamber the amount of  $\text{SO}_2$  oxidized was considerably greater, viz., 23 p.c. The intimate mingling and slight compression to which the gases are necessarily subjected in passing through the connecting tubes, is most probably the cause of the apparent anomalies in the oxidation noticed above. The authors suggest that a large number of small chambers may ultimately be found the most advantageous arrangement. In the investigations as to the uniformity of mixture of the gases, samples were collected simultaneously from the first chamber by the three sampling tubes II., III., IV. in the same vertical plane, in the side wall of the chamber. The analyses quoted show that the difference of composition of the gas collected at these points was very slight, in some cases, indeed scarcely apparent. Samples of gas were then taken simultaneously by the three tubes II., III., IV., at the side wall, and by three tubes inserted through the cover of the chamber at the point X., to the same depth respectively as II., III., IV. The following are the analyses of the samples so collected:

	Top		Middle		Bottom	
	Interior	Exterior	Interior	Exterior	Interior	Exterior
O .....	7.34	7.12	7.76	7.36	6.93	7.39
N .....	90.43	91.07	89.98	90.78	90.71	90.85
$\text{SO}_2$ .....	2.03	1.66	2.08	1.67	2.18	1.58
NO .....	0.68	0.08	0.08	0.10	0.10	0.10
$\text{N}_2\text{O}_5$ .....	0.12	0.08	0.11	0.09	0.07	0.08

The difference of composition of the gas at these different points is very small. The oxygen and sulphurous acid are slightly higher in the centre than near the sides, top, or bottom, whilst the nitrogen is slightly higher at the sides than in the centre. The composition of the gases at different levels in the same vertical plane, is too uniform to permit the assumption that the hot gases rise to the upper part of the chamber when introduced, and then in cooling gradually sink; it is more probable that the Glover gases rapidly mingle in the fore-part of the first chamber with the gases there present. The position of the openings of the pipes connecting the chambers therefore becomes a matter of minor importance. The reaction between  $\text{SO}_2$  and O appears to proceed more vigorously in the vicinity of the walls than in the middle of the chamber. The cause of this has yet to be determined.

IV. *Upon the Temperature of the Lead Chambers.*—The following observations were made as to the temperatures obtaining (a) when the chamber space per Ikilo. of sulphur was 1.3 cbm., (b) when the charge of pyrites was larger, and the chamber space per Ikilo. of sulphur 1.3 cbm. (a) The temperatures and strength of drips were recorded three times daily, the figures given are the average of 14 days' observations. In (b) the figures are the mean of 10 days' observations.

#### CHAMBER SPACE PER 1 KILO. S.

A. 1.8 Cbm. B. 1.3 Cbm.

Where Examined.	°C	Strength of Drips.	°C	Strength of Drips.
I. Tube from Glover .....	65°		75°	
XII. End wall of Chamber 1	60°	55°B	69°	55°B
Middle of Chamber 1 .....				
Bottom zone II .....	60°	52°B	71°	
Centre zone III .....	61.8°	53°B	73°	53°B
Upper zone IV .....	64.5°		76°	
V. Connecting pipe of Chambers 1 and 2 .....	45°	54°	57°	53°B
VI. Chamber 2 .....		53°B	41°	53°B
VII. Connecting pipe of Chambers 2 and 3 .....	27°		33°	51°B
VIII. Chamber 3 .....		53°B	24°	53°B
Outer air .....	9°		12°	

The temperatures observed oscillated between narrow limits, from 3° to 4°, being the maximum variation.

Even the temperatures of the Glover gases oscillated only between 62° and 66°, in spite of variations in the flow of acid. The influence of the atmospheric temperature was only appreciable in the third chamber, where the maximum variation was 10°, that of the temperature of the outer air being 15°. The proportion of nitrous fumes has a marked influence upon the temperatures. With strongly yellow chambers, there is a greater difference between the temperatures of the fore part and hinder parts of the system than when the chambers are worked light. The temperature at the top of the chamber was observed to be from 4° to 5° higher than that at the bottom. Experiments as to the cooling effect of the chamber walls were made by observing the temperatures at points 5, 10, and 25 cm. from the wall. When the temperature of the outer air was 19°, between 5 and 10 cm. from the wall, a cooling of 2° was observed, between 10 cm. and 25 cm. only of 1°. The temperature in the centre of the chamber was found to be 5° higher than at a distance of 25 cm. from the wall, or 8° higher than at a distance of 5 cm. This, however, can hardly be ascribed to a greater vigour of reaction in the centre, since there the gases contain more  $\text{SO}_2$  and O than at the sides. Some observations were also made of the temperatures obtaining in the fore part of the first chamber, where the oxidation of  $\text{SO}_2$  proceeds most vigorously. 10 m. from the end wall, the temperature of the chamber atmosphere was found to be from 80.5 to 83°; this is higher than that of the Glover gases, and considerably higher than that which is usually considered the most advantageous, viz., 55° to 60°. The authors consider that further experimental data are wanting before drawing conclusions as to the best temperatures at which to work the system. For a comparison of the temperatures obtaining under the application of water in the form of steam and in that of spray, two series of observations were made, with somewhat unexpected results. The following are the particulars:—(1) Application of steam. Temperature of air = 12°.

	Temperatures.	Excess over atmospheric temperatures.
Glover, I. ....	69°	57°
1st Chamber .....		
Bottom zone, II. ....	71°	59°
Middle zone, III. ....	73°	61°
Top zone, IV. ....	76°	64°
Connection between Chambers 1 and 2, V. ....	57°	45°
2nd Chamber, VI. ....	41°	39°
Connection between Chambers 2 and 3, VII. ....	36°	24°
3rd Chamber, VIII. ....	21°	12°

#### (2) Application of spray. Temperature of air = 24°.

	Temperatures.	Excess over atmospheric temperatures.
End wall, XII. ....	73°	49°
1st Chamber .....		
Bottom zone II. ....	73°	49°
Middle zone III. ....	75°	51°
Top zone IV. ....	78°	51°
Connection between Chambers 1 and 2, V. ....	68°	44°
2nd Chamber, VI. ....	53°	29°
Connection between Chambers 2 and 3, VII. ....	47°	23°
3rd Chamber, VIII. ....	31°	7°

With the application of spray the absolute temperatures are higher than with the application of steam, but their excess over the atmospheric temperature is less. The atmospheric temperature obtained during the experiments with water spray was certainly 12° higher than during the experiments with steam, but it has been shown in an earlier part of the paper that in the fore part of the system, the temperatures are not appreciably affected by external conditions. The authors do not consider that the substitution of steam by water spray can reduce appreciably the temperature of the chamber atmosphere.

—W. D. B.

*Preparation of Acetic Acid.* T. Göring. (German Pat. 28064, 1883.)

IN separating acetic acid from crude aqueous solutions the author takes advantage of its solubility in alcohols and ethers, which are insoluble in water, or comparatively so. For the exhaustive extraction of the acid a systematic treatment is required. According to the nature of the product required so may the extracting liquid, as well as its subsequent treatment, be varied. Thus it may be treated with bases, and the acid converted into salts, or it may be distilled, the acetic acid being left behind in the concentrated state, or it may be treated with water, whereby a dilute but purified acid is obtained.—C. F. C.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, CEMENTS.

*Slowly Setting Portland Cement.* C. Heintzel. (German Patent 28873, 84.)

INSTEAD of adding gypsum to the freshly ground cement, the inventor adds a solution of a soluble sulphate to the mixture before burning. Ferrous sulphate in the proportion of 0.5 to 2 per cent. is found to answer very well.

—C. F. C.

*Kilns for Burning Bricks, Tiles, &c.* Jacob Davies, Manchester. Eng. Pat. 2427, January 31, 1884.

THE invention consists of additions to a former patent, 2662, June 7, 1882, in which a square kiln was described, having fire-holes only on one side, and having air passages formed beneath the furnace flue, and communicating therewith by means of numerous air openings. According to the present invention the kiln is made of any suitable form, and fire openings are formed on both sides. The mouths of the fire-holes may be closed by doors, or may be bricked up, when the kiln is in operation. As in the former invention, air to support combustion is introduced through a suitable passage, which is covered in by bricks; small spaces are left between the bricks. The amount of air admitted to the air passage may be regulated in accordance with the degree of heat required in the kiln.—J. T.

## X.—METALLURGY, MINING, Etc.

*On the Present State of the Quicksilver Mines in Idria.* By Adolph Exeli. Chem. Zeit. 56, viii., 1008.

THE author remarks that many are still of the opinion that great waste of metal takes place in the process used in winning the quicksilver, and, as a matter of fact, until a few years ago, great waste did take place, but this has lately been greatly improved. Besides better constructed furnaces, and better condensing arrangements, the following are said to have influence on the results:—

1. An exact classification and separation of the ore.
2. A process of sampling as exact as possible.
3. An exact estimation of value of ore, and also of residues.

For the last-named purpose Eschka's method is universally employed (Chem. Zeit. 5, 735, 896, 979), instead of

as was formerly the case, throwing the residues from the iron retorts aside, and which contained a considerable amount of mercury, it has become possible to extract as nearly as possible all the metal. The introduction of prizes for those workmen who delivered residues which contain not more than a given percentage of mercury has had a very beneficial result. Wood and coal are used for heating purposes. In the year 1883 22,692,050 kg. of ore were worked, with an average of 0.95% Hg. In the whole working 226,876 kg. of mercury were won, and 490,750 kg. of Stupp (inferior metal or dross), with an average of 52,660 mercury. The loss in working was 6%. The cost of working 100 kg. of ore was 45 pf. (= 53 d.)

—F. T. S.

*An Improved Process for making a Black Pigment and Polishing Material.* Andrew French, North Shields. Eng. Pat. No. 7448. May 9, 1884.

THE improvements comprised in this patent consist in manufacturing sulphide of lead from "lead fume," a substance formed in the flues and condensers connected with lead ore smelting furnaces, by mixing with the "fume" a solution of an alkaline sulphide preferably sulphide of sodium. For every ton of "lead fume" a solution containing 3½ cwt. (anhydrous) commercial sulphide of sodium is required, this proportion being suitable for grey lead fume or blue fume. The black substance formed after mixing the above ingredients is separated by filtration, washed, pressed, dried and gently crushed. The material is then ready for use. The solution filtered from the black substance contains sulphite and hyposulphite of the alkali employed, and may be recovered by existing processes. The claim made by the patentee is:—Making a black pigment and polishing material by mixing sublimate of lead or "lead fume" with an alkaline sulphide in the manner described.

—G. H. B.

*Soldering Aluminium.* By Bourbonze. Chem. Zeit. 56, viii., 1008.

THAT so small an amount of aluminium should be in use is partly to be accounted for by the fact that until lately the metal either alone, or with other metals, was incapable of being soldered. The author states that this is easily accomplished by means of solders of tin and zinc, or of tin, bismuth, and aluminium. Those of tin and aluminium are to be preferred. According to the working which the soldered pieces have to undergo, the solders must have different compositions. If the metal is to be moulded an alloy of 45 pts. of tin and 10 pts. of aluminium should be used, which is malleable enough to withstand the hammering. Pieces which are not to be further worked are easily soldered with an alloy of tin and a less quantity of aluminium. The process is the same as in soldering tinplate, or it can be effected still better with the flame.—F. T. S.

*Purification and Decomposition of Sulphur, and Phosphorus-Slags, and Phosphates.* (D.R.P. 27, 106, 16 Sept. 1883. A. Frank Charlottenburg.) Chem. Zeit. 61, viii., 1093.

SLAGS or other phosphates in a finely divided state are treated with a concentrated solution of chloride of magnesium, by which the sulphur-compounds of the substance are decomposed with formation of sulphuretted hydrogen. In like manner the uncombined lime decomposes the chloride of magnesium, forming magnesia and calcium chloride, by which indirectly greater solubility is conferred upon the slag. Substances treated in this manner can then be further analysed by the usual methods.—F. T. S.

*Furnace for Reducing Zinc and other Metals.* G. W. v. Nawrocki, Berlin. (Communicated by J. Quaglio, J. Pintsch, and A. Lentz.) Eng. Pat. 4955, March 15, 1884.

THIS invention has for its principal object the conversion of the carbonic anhydride, produced by the reduction of



metals from their ores, into carbonic oxide, which may afterwards be used as fuel, and thereby the utilization of a portion of the heat generated by the consumption of coal in reducing furnaces.—J. T.

*Improvements in Smelting Ores and Metals, and in the Apparatus Employed therefor.* Samuel Richard Smyth. Eng. Pat. 5452, November 19, 1883. Received Provisional Protection only.

THE process consists in the application of "liquid hydrocarbon and liquid oxyhydro-compounds, also liquid alkali compounds, to the smelting of iron and other ores," etc.—W. G. M.

*Improved Processes and Apparatus for the Reduction of Iron and other Ores.* Alexander Melville Clark, Chancery Lane, Middlesex. Communication from J. B. Octave Thiéblemont, Paris. Eng. Pat. 5564, November 28, 1883.

IN this process the reduction is accomplished by means of gaseous instead of solid matter, and hence, owing to the non-reduction of oxides of silicon, phosphorus, sulphur, and arsenic, combined with oxide of iron or its gangues, under these circumstances, a very pure metal is to be obtained. The apparatus employed consists of calcining kilns, a reduction chamber, a re-heating and a melting furnace, and regenerators. The ore is fed into the kilns, which are at the top of the arrangement, where it is roasted by a portion of the waste gas from the reduction chamber, it is thence raked into the latter chamber, where the reduction takes place. The mass of spongy iron being then passed into an air-tight compartment cools down without oxidation; or it may be transferred directly to the re-heating furnace, in which it is agglomerated in a bath of fluid silicates, etc., washed with pure silicates, and worked into bars or otherwise treated. The metal may be converted into cast-iron or steel of high quality by adding a sufficient quantity of pig-iron to afford the necessary carburization; or the spongy metal may, with slight loss of iron, be melted in a blast furnace.—W. G. M.

*Improvements in the Manufacture of Bessemer Metal.* Henry Davis Pochin, Barnes, Surrey. Eng. Pat. 5568, November 29, 1883.

THE novelty consists in the lining of the converter with chromate of iron. Chrome ore, containing as little silica as possible, is shaped and set as a lining, about two feet in thickness, by means of fire-clay, or "basic lining" material, or a backing of one foot of fire-brick may be used to economise the chromate; or the converter may be lined in the usual way with the basic mixture, and while the latter is still plastic its interior should be studded throughout with masses of the chrome ore.—W. G. M.

*Improvements in the Production of Aluminium.* Gustav Baron de Overbeck, 23 Ryder Street, St. James'. Communicated by Hermann Niewerth, Hanover. Eng. Pat. 5756, December 15, 1883.

THE metal is to be separated by electrolysis from an aqueous or other solution of the aluminium salt of an organic acid (such as acetate), or of a mixture which, by double decomposition, will yield such salt. Or a mixture of a metallic chloride and aluminium sulphate may be employed, this yielding nascent aluminium chloride, which is at once split up into aluminium and chlorine.—W. G. M.

*Improvements in the Manufacture of Aluminium and Aluminium Alloys.* Samuel Pearce Wilding, 23, Rood Lane, London. Communication from Ludwig Grabau, Hanover. Eng. Pat. 5798, December 18, 1883. Received Provisional Protection only.

FOR pure aluminium a rich phosphor aluminium is prepared by melting phosphorus with aluminium, or by

fusing aluminium and phosphor salts with a reducing agent or otherwise. The product is to be crushed, mixed with alumina or argillaceous earth, covered with coal-dust, and heated to incandescence in a crucible. The phosphorus reduces alumina, yielding metal which mixes with that already present, and a slag of "phosphor-alumina." When cold the mass is crushed and the metal separated from slag, and re-fused with suitable flux. To produce any given alloy the phosphide of the required metal is substituted for that of aluminium; to increase the percentage of the latter metal the alloy may be re-phosphorised and heated with alumina. On a large scale a reverberatory furnace with a reducing flame may be used. "Manganic or carbonaceous metals" may be employed instead of phosphides, and the fluor compounds of aluminium in place of alumina.—W. G. M.

*Improvements in Apparatus for Grinding, Crushing or Reducing to Powder, Ores, Quartz or other like Substances.* Thos. Wm. Bassett Mumford and Robert Moodie, Victoria Docks, Essex. Eng. Pat. 5846, December 22, 1883.

THE material to be treated must first be broken to a suitable size. It is then fed by means of a hopper, fitted with an adjustable slide door to regulate the discharge, on to an inclined perforated, or reticulated plate with a continuous jogging motion imparted to it, over which pieces of iron and certain foreign substances may pass and thus be separated, and through which the ore (or other material) falls on to an inclined plane, and is thence distributed evenly over the grinding surface of crushing rollers. These rollers are preferably narrow and should have a high rotatory speed, the speed of the two rollers may be the same, or they may vary, so that a rubbing action is added to that of crushing; the rolls are kept clean by means of brushes pressed against them by means of a spring. After passing the rollers, the ore is elevated into a hopper containing two constantly vibrating inclined sieves placed one above the other, that part of the ore which will not pass the upper (or coarser) screen, is returned by a shoot to the crushing rollers; that which will pass this, but not the lower or fine mesh, falls into another spout communicating with a second pair of rollers; and that which passes through both is delivered by a third channel to the sack or receptacle for fine ore. The passage of the ore into one or other of these shoots may be adjusted by means of a valve or deflector. The ore which passes through the second pair of rollers is elevated to a second pair of sieves and undergoes treatment similar to that just described; a third, fourth, or any desired number of rollers and separating apparatus may be added until the whole of the ore has been crushed to the requisite degree of fineness; but the patentees have found that four pairs are generally sufficient. Tailings which have escaped crushing at the end may be elevated and returned by a shoot to the first hopper, and so worked through again. The whole of the sieves may be actuated simultaneously by eccentrics, cams, or tappets and rods connecting all the frames of the sieves.—W. G. M.

*Improvements in Recovering and Obtaining Tin from Tinned Metallic Surfaces.* Astley Paston Price, 47, Lincoln's Inn Fields. Eng. Pat. 5847, December 22, 1883.

THE metal to be treated is made the anode for a galvanic current in a bath of caustic alkali; when saturated, the resulting stannate to be electrolyzed for tin, or the tin to be obtained by other means, or the stannate itself to be used in the arts.—W. G. M.

*Improvements in the Process for Treating Zinc Ores.* Friedrich Carl Glaser, Berlin. Communication from the firm of G. v. Kramsta'sche Erben and Rudolf Wiester, Kattowitz. Eng. Pat. 5886, December 28, 1883.

ZINC ore mixed with coke or other reducing material is introduced into a species of cupola furnace of circular or

oval cross section, with tuyères arranged at one or different heights. Hot or cold blast may be used. The liberated zinc is at once reconverted into oxide, which is collected in dust chambers, connected with the flue, and preferably provided with hopper-like bottoms to facilitate the removal of the deposited dust.—W. G. M.

*Improvements in the Manufacture of Metallic Alloys or Compounds.* George Alexander Dick, 110, Cannon St., London. Eng. Pat. 5914, December 29, 1883.

THIS patent relates to the manufacture of alloys of copper, zinc, iron and phosphorus or manganese (or both). Phosphuret of iron, containing from 2 to 20 per cent. of phosphorus, and ferro-manganese or spiegeleisen, of, say 70 per cent. manganese, are added to a bath of molten zinc heated to about 1200° F., *i.e.*, as near to the volatilizing point as possible without actually reaching it; in this way 8 to 9 per cent. of the compound whose composition must be known, will be taken up by the zinc, or proportionately less if the temperature be lower. The saturated compound thus formed is, with or without a quantity of pure zinc, added to molten copper, the amount of copper in the resulting alloy to be from 45 to 75 per cent. Silicon, if present, increases the tenacity, but if the ferro-manganese contain more than 0.5 per cent. a proportionately larger percentage of pure zinc is employed.—W. G. M.

*Improvements Relating to the Hardening of Steel.* William Robert Lake, Southampton Buildings, Middlesex. Communication from Charles Reuben Childs, Hartford, Connecticut, U. S. Eng. Pat. 8244, May 26, 1884.

MARL, preferably in about the proportion of half a pound to the gallon, is added to the water or other liquid to be used for hardening the steel; it is well also to add two ounces per gallon weekly. A cream coloured, soft variety, probably rich in calcium carbonate, found in the Town of North East, New York State, answers the purpose well. The bath should stand some twelve hours before using.—W. G. M.

## XI.—FATS, OILS, AND SOAP MANUFACTURE.

*A Generally Applicable Method for the Examination of Fatty Substances.* By Baron Hübl. Dingl. Polyt. Journ. 253, 281, 1884.

THE author gives a brief *résumé* of the tests generally made use of hitherto. They may be divided into two classes, *viz.*, those depending upon the physical and those upon the chemical nature of the fats. Those referring to the physical nature are: (1) Determination of the specific gravity. This is of little value in the case of liquid fats, on account of their similarity in this respect, but in the case of solid fats and fatty and waxy substances gives valuable information; (2) Determination of the points of fusion and solidification. This test is of little value in the case of neutral fats or the mixtures of these with fatty acids which occur in commerce. Trustworthy information as to the nature and purity of a fat is much more likely to be afforded by the determination of the fusing point of the separated fatty acids; (3) The solubility of fats in acetic acid has recently been investigated by Valenta (compare Dingl. Polyt. Journ. 1884, 252, 296, and 253, 418), and the simple method devised by him gives useful results. There is unfortunately one drawback to any method based upon the degree of solubility, namely, that fats usually insoluble frequently pass into solution in the presence of a soluble fat; the presence of free oleic acid in rancid oils may therefore lead to discrepant results. The chemical methods of procedure having reference to the constitution of the fat are of two classes. In the one are to be placed those methods based upon the direct analytical estimation of a single constituent of the fat; such as Hehner's insoluble fatty acid method and processes for the determination of oleic acid and glycerol. In the second class

are Koettstorfer's saponification test, and the one devised by the author, and about to be described, depending upon the determination of the quantitative behaviour of the fat in a plain saponification process. Almost all fats contain members of three different groups of fatty acids, *viz.*, acids of the acetic series, of the acrylic series, and of the tetrolic series; the relative amount of the members of these groups in different fats is widely different, and determines to some extent the applicability of the fat to industrial uses. In their behaviour towards halogens, these three groups of fatty acids are clearly distinguished from each other. Under such conditions as preclude the possibility of substitution products being formed, the first group is quite indifferent to, the second group absorbs two atoms of, and the third group four atoms of the halogen, additive products being formed. Since the proportion and molecular weight of the unsaturated acids vary in different fats, the determination of the amount of halogen absorbed with formation of additive products will give results dependent upon the constitution of the fat. For several reasons the author has been led to employ iodine in preference to either chlorine or bromine (in reference to the application of bromine compare Mills and Snodgrass, *Journ. Soc. Chem. Ind.* 1883, p. 435, and Allen, 1883, p. 437). By itself, however, iodine reacts very slowly; on this account the author employs an alcoholic solution of iodine in conjunction with mercuric chloride, in the proportion of a molecule of the former to at least one of the latter. This solution reacts at ordinary temperatures with ease upon unsaturated fatty acids, either free, or combined as glycerides, to form chloro-iodo-addition products. From a practical point of view it is indifferent whether only iodine or chlorine, or if both, in what proportions, enter into union with the fatty acid, since the amount of halogen absorbed is estimated volumetrically and expressed in terms of iodine. For the performance of the test the following reagents are required: (1) Solution of iodine and mercuric chloride, called for brevity, *iodine solution*. This is prepared by dissolving 25 grms. of iodine in 500 c.c., and 30 grms. of mercuric chloride in 500 c.c. of alcohol of 95 p.c., free from fusil oil; the second solution is filtered if necessary, and the two solutions then united. This iodine solution should not be employed until it has stood for about 12 hours, as owing possibly to impurities in the alcohol it undergoes considerable reduction in strength during that period. It must, moreover, be always standardized immediately before or after use. (2) Solution of sodium thiosulphate containing about 24 grms. per litre. This is standardized by means of pure sublimed iodine, and on keeping does not alter in strength sufficiently for the test to be rendered inaccurate. (3) Chloroform, tested for purity by mixing with 10 c.c. of the iodine solution, allowing to stand for a few hours, and then comparing the amount of iodine with that of the original solution. If the chloroform has consumed no iodine it is fit for use. (4) A 10 p.c. aqueous solution of potassium iodide. (5) A recently prepared 1 p.c. starch paste. Of drying oils 0.2 to 0.3 grm., of non-drying oils 0.3 to 0.4, of solid fats 0.8 to 1.0 is weighed off, dissolved in 10 c.c. of the chloroform, and 20 c.c. iodine solution added. The amount of iodine must be such that after 1½ to 2 hours the solution may possess a dark brown tint. If that be not so in any case, 5 to 10 c.c. more of iodine solution are to be run in. The reaction is ended after about two hours' digestion at the ordinary temperature. From 10 to 15 c.c. of the potassium iodide solution are run in, and after diluting with 150 c.c. of water, the free iodine—part of which is in the aqueous, part in the chloroform solution—is titrated with the thiosulphate solution, the starch being added only when the yellow colouration has become very faint. The amount of iodine absorbed is calculated into units per cent. of the fat; this may conveniently be termed the iodine degree. A series of determinations with pure substances was made with a view of testing the method. It was found that in dealing with free fatty acids, the reaction was completed when only a small excess of iodine solution was present; a larger excess, however, must be present in working with fats, otherwise the results are too low. In presence of a sufficient excess of iodine, variations in the concentration of the fatty solution and in the



amount of mercuric chloride present do not affect the results. The reaction should be allowed to proceed for not less than two hours; digestion, however, for 48 hours does not influence the absorption. Four determinations of the iodine absorbed by chemically pure oleic acid gave figures varying from 89.8 to 90.5 p.c., the mean being 90.25 p.c., as compared with 90.07 p.c. indicated by theory. The author records in an extended table detailed results of his examination of several samples from different sources of a large number of oils and fats. The tabular statement here given presents the maxima, minima and average of the figures there recorded. The numbers referring to the saponification and to the solubility are derived from Valenta's published papers (*Dingl. Polyt. Journ.* 1883, 249, 271, 1884, 252, 297). The points of fusion and solidification of the fatty acids were determined by introducing the substance into a test tube of 7m.m. diameter, gently agitating with a thermometer, and noting at what point the whole contents became either quite clear or slightly cloudy. It will be observed that

of vegetable origin are characterized by the remarkable fact that their iodine degree does not correspond to the amount of oleic acid present but lies considerably higher. Especially is this apparent in oils from members of the eruciferous order. The addition of a mineral oil to any fat will naturally reduce the iodine degree of the latter and will at the same time affect the saponification test, and the solubility in acetic acid. From his experimental results the author draws the following conclusions:—Linseed oil is distinguished from all other oils by its high iodine degree, and any addition of a foreign oil must reduce that degree. The method is capable of detecting a falsification of 10 per cent. of cotton-seed oil, and 5 per cent. of a mineral oil. By boiling linseed oil, its iodine degree is lowered, but the fusing point of its fatty acids becomes higher. A linseed oil with iodine degree of 156 before showed 148 after conversion into varnish, whilst the points of fusion and solidification of the fatty acids became respectively 17.5 and 23. Between the different

Character of the Fat.	Name of the Fat.	Average Iodine degree.	Limits between which the Iodine Degree was found to Oscillate.	The Fatty Acids.		Mgms. of KOH required to Saponify 1 grm. of the Fat.	A Solution of the Fat in an equal amount of Glacial Acetic Acid (1.0562 sp. gr.) becomes turbid at °C.
				Fuse at °C.	Solidify at °C.		
I. Drying.	Linseed Oil .....	158	156 and 160	17.0	13.3	191.3	—
	Hemp Seed Oil ....	113	—	19.0	15.0	193.1	—
II. Drying.	Nut Oil .....	143	142 do. 144	20.0	16.0	196.0	—
	Poppy Oil .....	136	135 do. 137	20.5	16.5	194.6	—
	Pumpkin Seed Oil..	121	—	28.0	24.5	189.5	108
III. Moderately drying.	Sesame Oil .....	106	105 do. 108	26.0	22.3	190.0	107
	Cotton Seed Oil ....	106	105 do. 108	27.7	30.5	195.0	110
	Arachis Oil .....	103	101 do. 105	27.7	23.8	191.3	112
	Rape Seed Oil .....	100	97 do. 105	30.1	12.2	177.0	Insoluble.
IV. Non-drying.	Apricot Kernel Oil..	100	99 do. 102	4.5	0.0	192.9	114
	Almond Oil .....	98.4	97.5 do. 98.9	14.0	5.0	195.1	110
	Castor Oil .....	84.1	84.0 do. 84.7	13.0	3.0	181.0	Soluble in the cold.
	Olive Oil .....	82.8	81.6 do. 84.5	26.0	21.2	191.7	85 to 111.
	Olive Kernel Oil ....	81.8	—	—	—	188.5	Soluble in the cold.
V.	Bone Oil .....	68.0	66.0 do. 70.0	30.0	28.0	—	—
	Hog Lard .....	59.0	57.6 do. 60.0	—	—	195.9	—
	Artificial Butter....	55.3	—	42.0	39.8	—	—
VI.	Palm Oil .....	51.5	50.1 do. 52.4	47.8	42.7	202.2	23
	Laurel Oil .....	49.0	—	27.0	22.0	—	26.5
	Tallow .....	40.0	—	45.0	43.0	196.5	35
	Fat from Suint ....	36.0	—	41.8	40.0	170.0	—
	Cacao Butter .....	31.0	—	52.0	51.0	—	105
	Nutmeg Butter ....	31.0	—	42.5	40.0	—	27
	Butter Fat .....	31.0	26.0 do. 35.1	38.0	35.8	227.0	—
VII.	Cocoa Nut Oil .....	8.9	—	21.6	20.4	261.3	40
	Japanese Wax .....	4.2	—	—	—	222.2	—

the drying oils have the highest iodine degree, then follow the moderately-drying as cotton seed oil, then the non-drying vegetable oils, then the liquid and soft animal fats, and lastly the solid fats. All the oils members of group II. there are but small differences in the iodine degree. An addition of from 5 to 10 per cent. of an oil of group III. or IV. would be readily detected in nut or poppy oil, but not much less than 20 per cent.

of linseed oil. In group III. the iodine degrees are too nearly identical to establish the identity of the oil, but by reactions there will be little difficulty in arriving at the truth. As regards the absorption of iodine rape-seed oil is affected by the method of extraction and refining, the refined oil usually possesses an iodine degree 2 or 3 degrees below that of the raw product. A falsification with 15 per cent. of linseed oil may be detected with certainty. Castor oil possesses a very constant iodine degree, from 84.0 to 84.7, and is distinguished from all other oils by this, by the points of fusion and solidification of its fatty acids, by its saponification test and its ready solubility in alcohol and acetic acid. The admixture of foreign oils with this, or the falsification of an oil with castor-oil, may be readily detected. On account of the commercial importance, special attention was given to the examination of olive oil. Of the 20 samples—collected from the most widely differing sources—the absorption of iodine varied only within 3 degrees. The addition of about 5 per cent. of a drying oil, or 15 per cent. of cotton-seed, sesame, arachis, or rape-seed oil, may be established with certainty. Olive-kernel oil has very nearly the same iodine degree as olive oil, but it differs from the latter in possessing a dark greenish-brown colour and its solubility in alcohol of 95 per cent. and glacial acetic acid. The last named reactions will suffice to indicate the falsification of olive with a large quantity of olive-kernel oil. The oils extracted by carbon bisulphide from pressed marc resemble olive-kernel oil in their solubility, usually yield no solid elaidin, absorb 79 to 80 degrees of iodine, and are characterized by their dark colour and unpleasant smell. The amount of iodine absorbed by specimens from different sources of the other fats of groups V. and VI. appears to vary more than in the case of vegetable oils. Animal fats, as is well known, are liable to alteration in respect of their consistency and proportion of oleic acid, such being due in part to variety, age, and food of the animal whence they are derived. In the case of butter fat the absorption of iodine varies between the limits of 26 and 35. The iodine absorption of butter is from 15 to 20 degrees below that of a mixture of tallow and lard of the same consistency, this being most probably due to the presence in butter fat of the 7 or 8 per cent. of liquid fatty acids of the acetic series. The absorption of iodine by the different members of group VI. is too nearly alike to permit of different members of this group being distinguished from one another, since the iodine degree of each member may oscillate within comparatively wide limits. Where the nature of two fats in a mixture is known, their proportion may be determined approximately by the aid of the following formula:

$$X = \frac{100(I-n)}{m-n}$$

where X is the percentage of one fat,  $y$  that of the other, I the iodine degree of the mixture,  $m$  the iodine degree of the fat,  $x$   $n$  that of the fat  $y$ . The age of a fat, so long as great alterations have not taken place, does not affect its iodine absorption. If, however, an oil by the action of light and air has become thick and rancid and contains free acid, the iodine degree suffers considerable depression. The author believes his method will be found of value in the examination of soap, and in the determination of the value of oils—the constitution of which is yet uncertain—for technical purposes. Ethereal oils behave towards the iodine solution in the same manner as unsaturated fatty acids, and the above method may therefore afford a means of distinguishing between these and saturated compounds as certain hydrocarbons, phenol, and so forth. Since the above described alcoholic solution of iodine and mercuric chloride reacts upon unsaturated compounds, introducing halogens into their molecule under such conditions as preclude the possibility of a substitution taking place, it will probably be found of value in the synthesis of organic compounds.

—W. D. B.

*A Method for Testing Mineral Oils.* E. Valenta. Dingl. Poly. Journ. 253, 418, 1884.

THE question as to whether refined rosin oil can be employed in place of mineral oils for lubricating purposes has frequently been answered in the affirmative. In practice, however, it has been found that on exposure to the air rosin oil has a tendency to become sticky and viscid, and therefore bearings lubricated with it are apt to heat. It has also been found that continued exposure of this oil at higher temperatures to the action of certain reagents—the alkaline earths in particular—brings about the formation of a varnish-like substance with distinct drying properties, and that the addition of small amounts of this substance to mineral oils causes a marked increase in the viscosity of the latter. It is not impossible that an addition of rosin oil is sometimes made for the purpose of increasing the viscosity of mineral oil, but it is more probable that in the majority of cases this addition is to be regarded simply as a sophistication. The author has already pointed out (Dingl. Poly. Journ. 252, 296, 1884), that glacial acetic acid at 50° C. has only a slight solvent action upon mineral oils, whilst rosin oil is readily soluble in that reagent; in the two following tables the results obtained more recently by him are set forth:—

TABLE A.

*Showing the Solubility of various Mineral Oils in Glacial Acetic Acid (sp. gr. 1.0562 at 15° C.) at 50° C.*

No.	Name of Mineral Oil.	Sp. Gr. at 15° C.	Amount dissolved at 50° C., by 100grms. of Glacial Acetic Acid.	Amount dissolved at 50° C. by 10c.c. of Glacial Acetic Acid.	REMARKS.
1	Lubricating Oil.	0.9090	5.7648	0.6089	{ Clear, light yellow, strongly fluorescent inodorous oil.
	Ditto.	0.9090	5.7789	0.6104	{ Ditto.
3	Machine Oil (yellow).	0.9139	5.7333	0.6036	{ Clear, dark orange, inodorous, strongly fluorescent.
4	Spindle Oil (yellow).	0.9100	4.7778	0.5016	{ Light yellow, inodorous, strongly fluorescent.
5	Heavy Mineral Oil (thin).	0.9090	4.2810	0.1522	{ Oils of light to orange-yellow colour, of neutral reaction, inodorous, fluorescent.
6	Light Mineral Oil (thin).	0.8880	4.7009	0.1065	{
7	Viscous Mineral Oil.	0.9070	2.6729	0.2823	{ Dark brown, opaque, and with tarry odour.
8	Green Oil.	0.9105	6.4988	0.6819	{
9	Blue Oil.	0.9016	6.0170	0.6312	{ Dark brownish red, opaque, strongly fluorescent, and with tarry odour.
10	Vulcan Oil.	0.9259	3.3151	0.3525	{ Dark brown, opaque, non-viscid, strongly fluorescent, and with tarry odour.

Samples of 7 were obtained from Wagenmann and Co., Vienna. Samples 8 to 10 were from a laboratory collection.



TABLE B.

*Solubility at 50° C. of different Mixtures of Yellow Machine Oil (sp. gr. 0.9139), and Crude Rosin Oil (sp. gr. 1.0023), in Glacial Acetic Acid of 1.0562 sp. gr.*

No.	Percentage by Volume of Rosin Oil in the Mixture.	Amount of oil dissolved by 100 grams of Glacial Acetic Acid.	Amount of Oil dissolved by 10c.c. of Glacial Acetic Acid.	REMARKS.
		Grams.	Grams.	
1	0	5.7333	0.6056	The Crude Rosin Oil was obtained from Wagenmann and Co., Vienna. It had a dark brown colour, tarry odour, viscid consistency, and became resinous on exposure to the air.
2	25	7.3973	0.7796	
3	50	8.3633	0.8816	
4	75	12.5601	1.3237	
5	100	16.8782	1.7788	

For the performance of the test, 2c.c. of the suspected oil are mixed in a test tube with 10c.c. glacial acetic acid, the tube then being immersed for five minutes in a water bath, and repeatedly agitated during that period. The acid is filtered through a damp filter, the second third of the filtrate being collected. A weighed quantity of this is titrated with standard alkali, and the amount of acetic acid thus determined. The difference between the weights of the acetic acid taken and that found is the amount of oil dissolved. It appears from Table B that the solubility of a mixture of rosin and mineral oils is not proportional to the quantity of the former. The method, therefore, is not capable of yielding quantitative results. However, when combined with the determination of the rotary power (most rosin oils rotate the polarised ray very strongly, viz., about 30° to 40° in a 100mm. tube, whilst mineral oils are optically inactive), and with that of the amount of iodine absorbed, according to Hübl's method (the author having found that 1grm. of mineral oil seldom absorbed more than 140mgrms. of iodine, whilst the absorption by rosin oils was found to vary between 430 and 480mgrms. per 1grm. of oil), the author considers that there are very few cases where this method will not detect the sophistication of a mineral oil with rosin oil.—W. D. B.

together. Red lead, and lastly quadroxalate of potash which possesses the property of destroying both animal and vegetable life. A dryer such as oxide of mercury or sugar of lead is used with the above. The whole of the ingredients are ground together in a dry state, and afterwards into a paint with a varnish composed of two portions, prepared separately and mixed in the cold; consisting of 1, shellac dissolved in methylated spirits; 2, rosin, boiled linseed oil, turpentine and spirit of tar. Any suitable colouring matter such as venetian red may be added to the composition.—G. H. B.

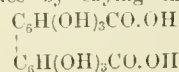
## XIII.—TANNING, LEATHER, GLUE AND SIZE.

*Improvements in Waterproofing Leather.* John Cove and W. P. Sherwood. Eng. Pat. 9589, June 30, 1884.

THE leather, or leather-board is thoroughly dried in a warm chamber till it ceases to lose weight, and is then immersed in a mixture of about one part of vaselin with three or four parts of paraffin, melted at a temperature of about 113° F. until bubbles cease to rise from it.—H. R. P.

*On the Behaviour of Tannin and Tannic Acid from Oak-Bark towards various Reagents.* By C. Etti, Ber. Chem. Ges., 1884, p. 1820.

THE reactions which take place when tannin from nut-galls and tannic acid from oak-bark are subjected to the influence of the same reagents are compared, and the great difference existing between these two bodies is pointed out. Thus on boiling tannin from nut-galls with dilute sulphuric acid, water is taken up by the former and gallic acid is produced, whereas tannic acid from oak-bark under the same conditions loses water and forms an anhydride but no gallic acid. Pure tannic acid from oak-bark, producing with lead acetate a yellow precipitate, is at a temperature of 130 to 140° converted into an anhydride, producing with lead acetate a reddish brown precipitate; tannin from nut-galls remains unaltered even at 200°. The body last mentioned forms acetyl compounds of definite composition: acetyl compounds of tannic acid from oak-bark, in which the tannic acid is combined as such (*i.e.*, unaltered) with the  $C_2H_3O$  molecule cannot be obtained. Tannin from nut-galls, when boiled with aqueous ammonia in an atmosphere of hydrogen, yields in almost the theoretical quantity, gallamide,  $C_6H_2(OH)_3.CO.NH_2$  and acid ammonium gallate in well formed crystals, whilst tannic acid from oak-bark forms an amorphous brown compound. These facts prove that tannin from nut-galls and tannic acid from oak-bark are not identical, and that the former compound cannot be a constituent of the latter. The author draws attention to the analogy in the reaction of ammonia upon tannin and lactic anhydride; and, referring to the results obtained by H. Schiff in his researches on the acetyl compounds of tannin, concludes by saying that the formula—



*Improvements in the Production of Compounds capable of being employed for the Destruction of Insects upon Vegetation, as an Antiseptic, and for Cleansing, Disinfecting, and for Lubricating Purposes.* W. G. Little, Conisboro. Eng. Pat. 275, January 2nd, 1884.

THIS is a patent relating to the production and use of compounds obtained by saponification with potash of heavy and light resin oils. By saponification the oils become miscible with water, in which state the liquid can be used as a means of destroying insects upon vegetation, as an antiseptic, for cleansing or disinfecting purposes, or as a lubricant.—G. H. B.

## XII.—PAINTS, VARNISHES, AND RESINS.

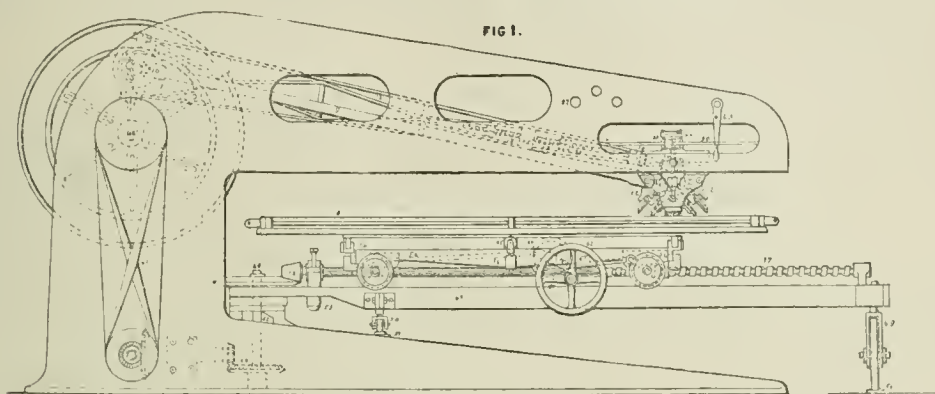
*An Improved Anti-fouling Composition for Coating Ship's Bottoms and Submerged Metal Structures.* J. Pickering and H. E. A. Saale, London. Eng. Pat. 10457, July 22, 1884.

THIS invention has for its object an anti-fouling composition to resist the action of sea water, and prevent the adhesion of animal and vegetable growths. The inventors specify the materials used in the preparation of the mixture, and give their reasons for employing those materials as follows:—Carbonate of baryta—fatal to vegetable life; arsenite of baryta—fatal to animal life. Powdered oyster shells or their equivalent repel oysters and other shell fish, and possess besides the peculiar property of repelling vegetable growth. Oxide of zinc is employed to resist the action of the sea water, and is valuable in admixture for preventing the formation of heavy sediments when the several ingredients are put

i.e., digallic acid hitherto assigned to tannic acid from oak-bark, and tannin from nut-galls, cannot any longer be upheld.—F. M.

*Improvements in Machinery for Dressing Leather.* Sam Haley. Eng. Pat. 4677, March 2, 1884.

THIS is a modification of the Fitzhenry or Jackson scouring machine, in which the tools are lifted off the work during the return stroke by a compound connecting rod operated by an eccentric (13) carried by the crank pin (16). The tool holder carries four or eight stones or sleakers, and is provided with spiral springs and india-rubber cushions to relieve the pressure on uneven parts of the leather. Two tables (of which only one is shown)



are provided, which are supported on a pivot (48) and upon wheels (49) resting on a rail of quadrant form, so that they are capable of radial motion. They may also be moved automatically from or towards the centre by a screw (37) driven in either direction by belts (41) and gearing (38-39). The tables are intended to be used alternately to save time.—H. R. P.

## XV.—SUGAR, GUMS, STARCHES, ETC.

*Improvements in the Refining of Sugar and Apparatus therefor.* Arsène de Marrousseno. Eng. Pat. 10424, July 22, 1884.

THIS patent relates to the production of pure white sugar in the form of lumps, cubes or tablets. The author claims that it differs from those of former inventors, who have sought to expedite such production, in the following respects. Former methods have always involved the placing of the syrup brought to the requisite consistency in moulds to induce crystallization by cooling, and afterwards the separation of the syrup from the crystals in specially constructed centrifugal machines. In the present process the preliminary crystallizing operations are entirely done away with. The syrup boiled to the requisite consistency is filled directly into the centrifugal machine, and entering the same in a hot, sticky condition it moulds itself under the pressure of the centrifugal force, before undergoing the action of steam, air, water, clarifying liquor or other agents designed to assist the refining process. By this method the whole operation is completed in a much shorter time.—A. J. K.

*Improvements in India-rubber Hose or Flexible Tubing.* J. B. Harris, Edinburgh. Eng. Pat. 6066, April 7, 1884.

THIS is a patent to substitute brass wire gauze for the ordinary web of cotton or flax used in making india-rubber hose or flexible tubing. The brass gauze is coated with a preparation of india-rubber, in the same manner as with textile materials, and when so coated is cut up into diagonal strips of suitable widths of which the hose is made up as with rubber coated textile materials. The

advantage claimed as an improvement over the ordinary cotton or flax webs is that the brass web is unaffected by heat.—G. H. B.

*An Improved Method of Producing White Crystalline Sugar from the Raw Sugar Mass.* Carl Steffen. Eng. Pat. 11347. August 16, 1884.

THE "massesquites" obtained from the vacuum pan is at once desiccated by means of hot air in revolving cylinders, and the dried mass is then lixiviated by a solution of pure sugar specially prepared until the impure syrups formerly in the "masse" have been completely removed and only pure sugar remains in the lixiviating apparatus.—A. J. K.

*Improvement in Cane Juice Clarifiers.* George Fletcher Poplar. Eng. Pat. 11439, August 19, 1884.

THIS invention relates to improvements in the arrangement of steam clarifiers and steam heating pans for cane juice. The usual arrangement of such vessels consists of two plain iron or copper pans fitted one inside the other in such a way that the space between may be used as a steam jacket. The improvement contained in this patent consists in making the inner vessel of corrugated iron. The corrugations give about 25 per cent. more heating surface, and the corrugated plates are stronger weight for weight than plain flat ones, hence thinner plates can be used. Such an arrangement also obviates to a great extent the necessity for steam coils within the pan, and thus gives increased facility for cleaning the same.—A. J. K.

*Improved Apparatus for use in treating Wood and other Ligneous Matter by means of Hydrochloric Acid Gas in order to obtain Glucose therefrom.* A. M. Clark. Eng. Pat. 11557, August 22, 1884.

THE apparatus described by the inventor may be used in any of the various processes for the treatment of ligneous matters by hydrochloric acid gas, but is more particularly adapted to the Danjéville process for converting wood cellulose into glucose and alcohol. The apparatus consists of four distinct parts. (1) For the treatment of the ligneous matter by the gas. This apparatus consists of a long leaden cylinder completely closed and supported outside by a wooden frame work and iron rings. The wood is introduced through suitable manholes, and the gas by leaden pipes through the hollow journals at the ends of the cylinder. The cylinder is caused to rotate slowly and is kept at a low temperature by a constant stream of cold water flowing outside. About 14 hours serves for the complete solution of the cellulose. The gas is now turned off and a little water or liquid introduced to wash the cylinder which is further revolved for half an hour. A plug is now withdrawn and the mass allowed to fall into asbestos packed filters. The clear filtrate from these which may be 20° B. to 22° B. is moved to depositing vats, and the cylinder now further washed out; the thin wash liquors are used in succeeding



operations. (2) Apparatus for removing hydrochloric acid from the concentrated liquids. This apparatus consists of a vacuum pan and a tubular condenser made either entirely of lead or of cast-iron lined with lead throughout. The liquid is evaporated till the glucose contains only about 7 per cent. of acid. The acid recovered is about 15° B., and is used for producing the gas for another operation. This is done in apparatus (No. 3). This consists of a domed cylinder inside of which is an inner cylindrical vessel containing strong sulphuric acid and heated inside and out with steam coils. Into this acid the weak hydrochloric acid is allowed to fall gradually, and the hydrochloric acid gas is at once evolved, leaving its water with the sulphuric acid. This apparatus is also entirely constructed of lead or of iron lined with lead. The fourth and last set of apparatus consists of an evaporating chamber and a condenser for concentrating the sulphuric acid from the last apparatus, and it is so arranged that these two processes work together and continuously, the weak sulphuric acid as it accumulates in No. 3 apparatus constantly flowing over into the evaporating chamber of No. 4.—A. J. K.

## XVI.—BREWING, WINES, SPIRITS, Etc.

*On the Gluten-precipitate in Beer.* Chem. Zeit. 60, viii., 1078.

MICHEL concludes from a number of small experiments that the so-called gluten-precipitate in beer is not, as has hitherto been supposed, a nitrogenous compound, but is a resinoid substance. It is especially characteristic that such beer, when shaken with ether, becomes clear, whereas, when the latter is evaporated, an aromatic substance remains, which contains white crystals. These crystals appear to be identical with Lerner's "Hopfenbittersäure."—F. T. S.

## XVIII.—SANITARY CHEMISTRY, DISINFECTANTS.

*Purification of Waste Waters.* Chem. Zeit. viii., 56, 1008.

J. KÖNIG has made experiments with a view to deodorizing the waste waters of tanneries and slaughter houses by atmospheric oxidation. By means of a tinued iron wire net the greatest possible amount of surface was given to the liquid, which was allowed to flow over the net. A very intense oxidation took place as the following experimental numbers show. The experimental water contained—

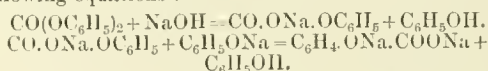
	Before	After
Oxygen.....	3°0c.c.	9°0c.c.
Sulphuretted hydrogen .....	20'4mg.	0'9mg.
Sulphuric acid .....	48'6mg.	72'0mg.

Suspended organic matter should first be separated in a settling tank, or the liquid filtered through sand. The height of the net should be 4.5m. for 20mg. H<sub>2</sub>S per litre. The breadth should be 1m. for every 12 litres of liquid per minute.—F. T. S.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Synthesis of Salicylic Acid.* Dingl. Poly. Journ., 253, 302, 1884.

It has been found by the Chemische Fabrik, vormals Hoffmann and Schoetensack (Ger. Pat. 27609, June 30, 1883) that Hentschel's process does not give a good yield of acid owing to the formation of a basic salicylate of soda and simultaneously with that of a correspondingly large amount of phenol. Better results are obtained by fusing together equal molecular weights of diphenyl carbonate, sodium hydrate, and sodium phenate. The reaction which takes place may be represented by the following equations:



—W. D. B.

*The Occurrence of Kino in Kino from Malabar.* C. Ettl. Ber. 17, 2241-2244.

THE author has always been able to obtain from kino, coming from different localities, the same crystalline body, kinoin (Ber. 11, 1879); but Krenler, and also Bergholz, have failed to isolate it. It is, however, shown that Krenler's experiments were very deficient, and that Bergholz did actually obtain kinoin, but in an impure condition.—H. B.

*Brucine.* A. Hanssen. Ber. 17, 2266.

BRUCINE heated with hydrochloric acid yields methylchloride, and a crystalline body melting at 284°; it is soluble in alkalis, and is reprecipitated by the careful addition of acids. Several reactions, and the analysis of this compound and its platinum double salt, are given. The work is being continued.—H. B.

*On Juglone.* By A. Bernthsen. Ber. Chem. Ges. 1884, p. 1945.

VOGEL and Reischauer obtained from walnut [juglans regia] husks a substance which they called nucin or juglone. This body has again been made the subject of an investigation by the author. Juglone crystallizes from ether or petroleum spirit in yellow needles, which in ammonia and dilute alkalis dissolve with a beautiful purple colour. Its percentage composition and properties go to prove that it is an oxy-naphtho-quinone C<sub>10</sub>H<sub>6</sub>O<sub>3</sub>. Subjected to dry distillation with an excess of zinc dust it yields naphthalene. It can be easily reduced by means of zinc and hydrochloric acid or stannous chloride to a colourless compound possessing the character of a phenol. By mixing a solution of juglone with one of copper acetate, a copper compound, having the formula (C<sub>10</sub>H<sub>5</sub>O<sub>3</sub>)<sub>2</sub>Cu, is formed. Aniline dissolved in glacial acetic acid combines with juglone, same as with ordinary oxy-naphtho-quinone under formation of a red compound, soluble in alcohol. Juglone crystallizes unchanged from dilute nitric acid, whereas an acid of 1.15 s.g. converts it into a soluble acid.—F. M.

## XXI.—EXPLOSIVES, MATCHES, Etc.

*Improvements in the Manufacture of and Contributions to a Knowledge of Explosive Compounds.* Dingl. Poly. Journ. 252, 153, 1884.

A COMMITTEE of the Société de l'Industrie Minérale have recently investigated, by practical trials, the comparative efficiency of ordinary Kieselguhr dynamite and Michalowski's blasting powder ("Bergmannspulver,") which consists of 45 parts of bran, 50 parts of potassium chlorate, 5 parts of manganese binoxide. The work performed by the latter appeared to be about equal to that performed by the former explosive, but the results cannot be considered conclusive. Michalowski's powder is bulky, and requires to be well rammed in the bore-hole. The liability to explosion by percussion of a mixture containing so large a proportion of chlorate constitute a serious objection to its employment. The Société la Pancelastie propose as new explosives certain mixtures of tar, asphalt or the like, with salts rich in oxygen. The following are specimens:—

I.	
10 parts .....	KClO <sub>3</sub> or NaClO <sub>3</sub>
5 " .....	Pb(NO <sub>3</sub> ) <sub>2</sub>
5 " .....	KNO <sub>3</sub> or NaNO <sub>3</sub>
2½ " .....	Hard pitch
2½ " .....	Tar

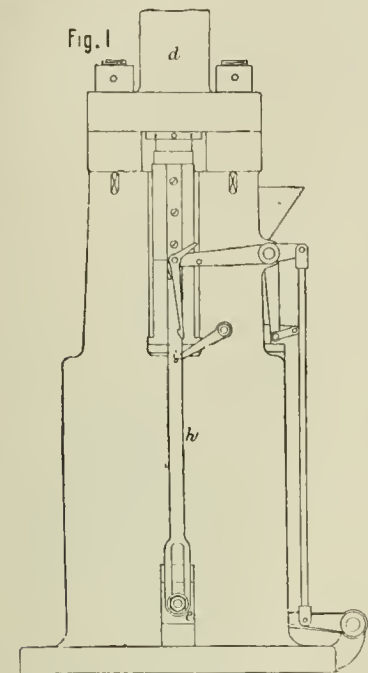
to which may be added

1 part .....	KMnO <sub>4</sub>
2 " .....	NaHCO <sub>3</sub>

II.	
80 parts .....	KClO <sub>3</sub>
10 " .....	Hard pitch
10 " .....	Tar.

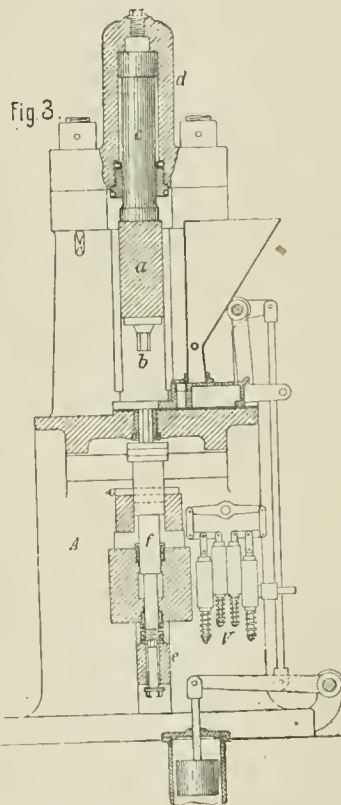
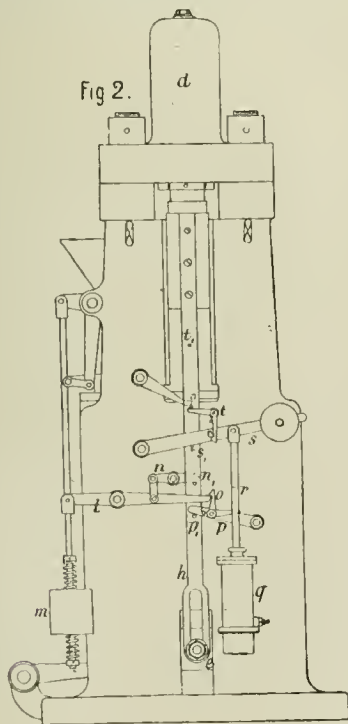
III.	
40 parts .....	Pb(NO <sub>3</sub> ) <sub>2</sub>
40 " .....	KNO <sub>3</sub>
10 " .....	Hard pitch
10 " .....	Tar.

If in III. the potassium nitrate be replaced by nitrate of lead, the mixture, it is stated, may, like dynamite or gun-cotton, be exploded in an unconfined space by means of a detonator. A new self-acting press for prismatic powder has been devised by Max Hasse & Co. (Ger. Pat. 24903, May 11, 1883), and is represented in



required and dispersed by a second lens. The spontaneous decomposition of blasting gelatine under ordinary climatic conditions has recently been observed in America by C. E. Munroe (*Journal of the American Chemical Society*, 1884, 13). A small quantity of blasting gelatine was kept in a room at the common temperature. After a year the substance was observed to be evolving nitrous fumes, which had attacked the wrapping of paraffined paper. The bulk of the substance had increased, and the exterior of the paper envelope was covered with small crystals. Thrown into water, the mass became friable, the odour of camphor disappeared, the water taking a yellowish colour and acid reaction. The aqueous solution, which contained traces of nitrous but no nitric acid, was filtered and evaporated, when a considerable quantity of oxalic acid crystallized out; the mother liquor was concentrated upon the water bath and was found to contain a sugar-like substance, which gave

figs. 1 to 5. For the illumination of powder works, the electric light has been successfully applied. In the sifting and granulating houses at Waltham Abbey, in-

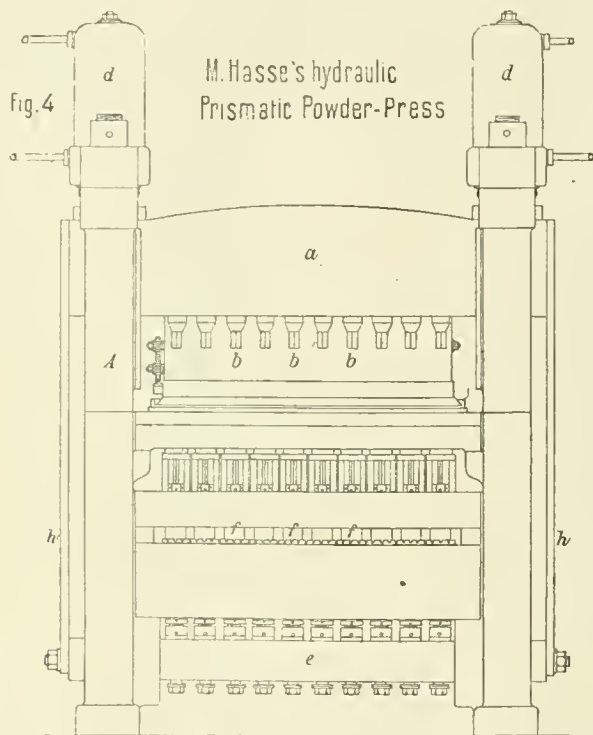


the glucose reaction with Fehling's solution. Careful examination failed to detect any glycerine, nitro-glycerine, or gun-cotton. This decomposition was probably owing to the presence of free acid in the nitro-cellulose, and it would hardly be just to draw conclusions from this case as to a specific liability of blasting gelatine to spontaneous decomposition. Cases of spontaneous decomposition of blasting gelatine have also been observed by Abbot, who has recorded them in the appendix to his report upon submarine blasting operations (New York, 1883.) An explosion which occurred at the James Watt Dock, Greenock, owing to a cartridge of tonite being forced into the bore-hole with a metallic rammer, and the explosion, which took place at Chilworth Powder Works caused by a copper tool being employed to loosen the cake from the incorporating mill, alike demonstrate the danger attendant upon the use of metallic tools. The work performed under water in a horizontal direction by tonite, gun-cotton, and the so-called rackarock has recently been the subject of experiments by General Abbot (*Engineering and Mining Journal*, 1884, 26). Both the gun-cotton and tonite were manufactured in

candescant lamps are now fitted. In the Pulverfabrik Oehtha the rays from an arc light are brought parallel by passing through a lens, conducted in tubes where



San Francisco. The former contained 89.6p.c. of tri-nitro-cellulose, 10.4p.c. of di-nitro-cellulose; the tonite contained 52.5p.c. of gun-cotton and 47.5p.c. of barium nitrate. Rackarock is one of Sprengel's explosives, and is supplied for use in two parts, neither of which possesses explosive properties. The one part consists of cartridges of potassium chlorate dyed red, the other of nitro-benzene, in which, when required, the first are immersed until their weight has increased by about one-third. The explosive so prepared forms a hard mass of 1.7sp. gr., not easily brought to explosion under a hammer, and is not completely detonated even with a charge of 1.5grms.



## XXII.—GENERAL ANALYTICAL CHEMISTRY.

*Apparatus for Extraction of Liquids with Ether, Petroleum Spirit, Etc.* H. Schwarz, Zeitschr. Anal. Chem. 23, 368.

IN the preparation of organic substances it is frequently necessary to extract by shaking up with ether. This mode of extraction is inconvenient, for it causes great loss of ether, and sometimes leaves the fluids in a state of emulsion. For quantitative work the method has either to be abandoned or carried out with extreme care.

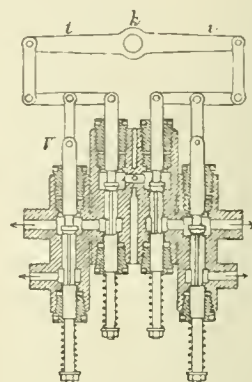


Fig. 5.

of fulminate of mercury unless very strongly confined. If fired into with a rifle, a cartridge of rackarock is merely inflamed. The practical results obtained from the above-mentioned explosives, and also from several others submitted to examination are here detailed:—

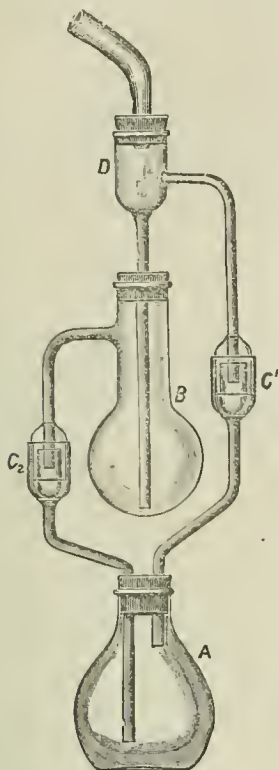
Name	Percentage of Nitro-glycerine	Work in a horizontal direction That performed by No. 1 Dynamite=100
Dynamite No. 1	75	100
Gun-cotton	—	87
Dualine	—	111
Rendrock	20	78
"	40	94
"	60	95
Dynamite No. 2	36	83
Vulcan Powder No. 1	30	78
" No. 2	35	82
Mica Powder No. 1	52	83
" No. 2	40	62
Nitro-glycerine	100	81
Hercules Powder No. 1	77	106
" No. 2	42	83
Electric Powder No. 1	33	69
" No. 2	28	62
Designolle's Chlorate Powder	—	68
Brugers	—	81
Blasting Gelatine	89	117
Tonite	—	81
California Gun-cotton	—	87
Rackarock	—	86

The addition of picric acid in quantity of from 12 to 16p.c. of the nitro-benzene employed, was not found of any advantage in the preparation of rackarock. A pyrographic method for testing gunpowder is described by C. E. Munroe (*Journal of the American Chemical Society*, 1884, 7).—W. D. B.

The apparatus shown in the figure (p. 649) is well adapted for such extractions: it is a modification of the form usually employed in the case of solids. Instead of shaking up the liquid with ether, the latter is allowed to ascend in a stream through the liquid. *A* is a small distillation-flask which contains ether, and is provided with a doubly-perforated cork. *B* is the extraction flask, and is provided with a side-tube bent downwards: its neck is closed by a cork, through which the funnel-tube *D* passes, reaching down to the bottom of *B*. *D* is also provided with a side-tube. The condenser is connected with *D* by means of an adapter. *B* and *D* are connected by means of mercury-cups *C*<sub>1</sub> and *C*<sub>2</sub> with the tubes from the flask *A*. *A* is gently heated up on a water-bath: the ether vapour ascends through *C*<sub>1</sub> and *D* into the condenser. The condensed ether flows back through *D* under the liquid, through which it ascends, and collects in the neck of *B*, finally flowing over by the side-tube *C*<sub>2</sub> into *A*. The operation does not require any watching, but should be allowed to proceed until nothing further is extracted. Water is poured carefully into *D*, so as to drive all the ether into *A*, *B* is emptied and washed out: the apparatus is now ready for a fresh operation. This process gives very satisfactory quantitative results. The extraction with fluids heavier than water, as carbon bisulphide and chloroform, is not effected with this apparatus, but the following results with ether testify to the value of the apparatus:

1. —2grms. of crystallized resorcinol dissolved in 50c.c. of water, were taken, and extracted with ether in this apparatus. The ether left, after extraction, 1.975grms. of resorcinol, and hence there was scarcely any loss.

- 2.—A resorcinol melt from 5grms. of potassium benzene disulphonate gave, after acidifying and extraction in the apparatus, 1.68 instead of 1.75grms. of resorcinol.
- 3.—0.3105g. alizarin, dissolved in alkali, acidified and extracted, gave a residue of 0.301g. alizarin.



- 4.—50c.c. of milk, rendered alkaline and extracted with ether, gave 2.593% of fat. A sample of the same milk evaporated down on a weighed sand-filter, and treated in an ordinary extraction-apparatus, gave 2.63% of fat.
- D. E. J.

*The Separation of Arsenic from Tin and Antimony.*  
F. Hufschmidt. Ber. 17, 2245.

FISCHER'S method of separating these metals consists in converting the arsenic into the form of arsenic trichloride by means of ferrous chloride and hydrochloric acid, and subsequent removal by distillation (See Ann. Chem. Pharm. 208, 182). The removal of the arsenic is, however, very slow; but by saturating the liquid with hydrochloric acid gas, and then distilling in a rapid current of the gas, every trace of the arsenic is removed before one fifth of the solution has passed over. Owing to this extreme volatility, Fischer's apparatus requires modifying by connecting with the receiver a Woulff's bottle of about 900c.c. capacity, half filled with water, or caustic soda water solution; the connecting tube should be wide. Numerous test analyses are given proving the great accuracy of the method.—H. B.

*Paratoluidine Sulphate as a Reagent for Nitric Acid.*  
Antonio Longi. Zeitschr. Anal. Chem. 23, 350.

WHEN nitric acid is added to a solution of paratoluidine in sulphuric acid, a blue coloration is produced, which changes to violet, red, and finally yellowish-brown. Rosenstiehl and Lanth have employed this reaction for showing the presence of paratoluidine; it also gives good results when used for the detection of nitric acid. A few drops of a solution of paratoluidine sulphate are added to the liquid containing the nitrate; an equal volume of

sulphuric acid is poured in, so that two layers are formed: at their common surface a red ring is observed, which finally becomes dark yellow. If chlorates, bromates, iodates, chromates, or permanganates are present, a blue (instead of red) colour is produced, and this is so intense that it marks the reaction of the nitrates, if these latter are only present in small quantity. Aniline sulphate gives no coloration; but if a mixture of the salts of both bases be employed the reaction is found to be more delicate. A solution of "*fuchsine aniline*" in dilute sulphuric acid can therefore be employed with advantage in place of pure paratoluidine. By means of this reaction the presence of nitric acid can be shown in a liquid containing only one part in 32,000. It may be observed that the delicacy of the ferrous sulphate reaction varies with the mode of manipulation. If the liquid to be tested is mixed with an equal volume of  $H_2SO_4$ , allowed to cool, and a concentrated solution of  $FeSO_4$  added, one part in 2,000 can be detected; but if an equal volume of  $H_2SO_4$  is added to the liquid (without mixing) and a few crystals of  $FeSO_4$  are thrown in, 1 in 8,000 can be detected. Brucine was long considered to be the most delicate test for nitric acid; by its means 1 part in 256,000 can be recognised. By far the most delicate reagent for nitric and nitrous acids is diphenylamine, which should be used as follows:—A few drops of diphenylamine sulphate are added to the liquid under examination, and a layer of concentrated sulphuric acid poured in; even when the liquid only contains 1 part in 1,500,000, a distinct blue coloration can be observed. Although paratoluidine is less sensitive than brucine and diphenylamine, it is preferable to these reagents because it does not give the reaction with chloric, bromic, iodic, or nitrous acids. Nitrous acid gives with paratoluidine a yellow colour, which after some time changes into red, owing to the partial conversion of nitrous into nitric acid. In order to detect nitric acid in presence of a large quantity of nitrous acid, the latter should be destroyed by means of urea. An excess of urea is added to the solution, and then, gradually, acetic acid, until no more gas is evolved in the cold: the liquid is evaporated to dryness on the water-bath, the residue dissolved in water and tested for nitric acid.—D. E. J.

*Valuation of Calcium Tartrate.* L. Weigert. Zeitschr. Anal. Chem. 23, 357.

THE value of cream of tartar, as well as of calcium tartrate depends entirely upon the percentage of tartaric acid which it contains, and it is immaterial whether this be determined as calcium tartrate or acid potassium tartrate. The method of analysis usually adopted is Soxhlet's. The percentage of  $CaCO_3$  contained in the sample is first found; a portion is then heated, so as to convert the tartrate into carbonate, and the total quantity of  $CO_2$  evolved on adding  $HCl$  is determined. It should be observed that the carbonic acid cannot be determined volumetrically, and that a second analysis is always required as a check. The author proposes the following rapid and accurate method which depends upon the fact that calcium tartrate and  $K_2CO_3$ , when boiled together, are converted into neutral potassium tartrate and  $CaCO_3$ . The liquid is filtered and evaporated down to a small volume: sufficient acetic acid is added to convert into cream of tartar, and this is washed with alcohol, and titrated. A solution of potassium carbonate is made by dissolving 100g. pure  $K_2CO_3$  in 1 litre of water; 5.5c.c. of this corresponds to 1g. calcium tartrate. 5g. of the latter are taken for the analysis, so that 30c.c. of the above solution should be employed. Boil in a flask in the water-bath for an hour, filter into a porcelain basin (taking care to wash very thoroughly); evaporate down to about 5c.c., and add 5c.c. of concentrated acetic acid. Warm for a few minutes in the water-bath; add 100c.c. of alcohol (strength=90% by volume) and allow to stand for a few hours. Filter and wash with 100c.c. of 80% to 90% alcohol. For the titration, potash solution should be used of such strength that 1c.c. corresponds to 0.02508g. cream of tartar. When 5g. of calcium tartrate are taken for analysis, and the results are calculated out as  $KC_4H_4O_6$ , 0.33% must be added. It has already been stated that the



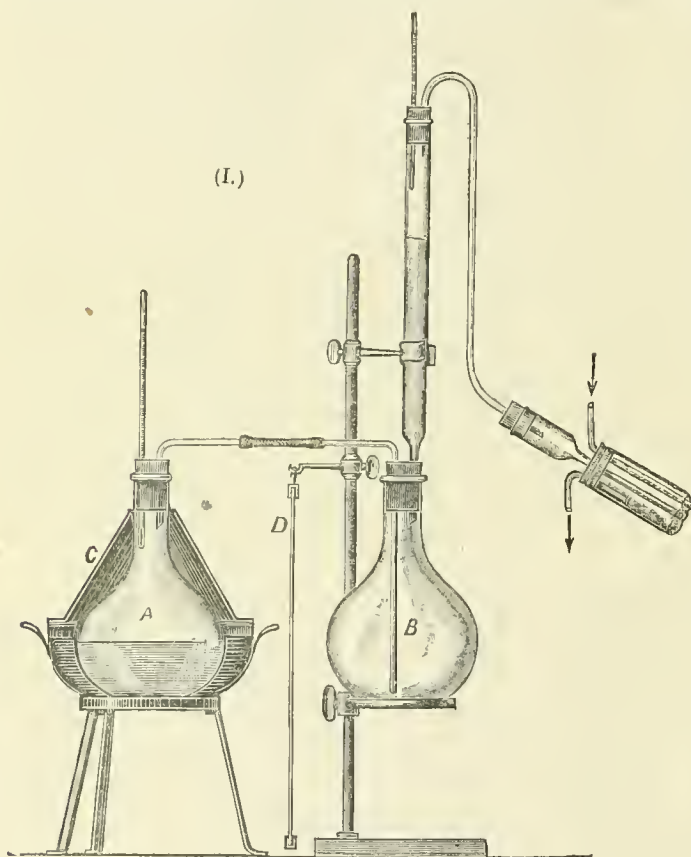
sample should be tested for  $\text{CaCO}_3$ , and that this cannot be determined by titration. The author recommends as a rapid and accurate method, that described by Mohr (Titrimethode; 4th edn. p. 551).—D. E. J.

*Apparatus for Fractional Distillation.* L. Weigert. Zeitschr. Anal. Chem. 23, 365.

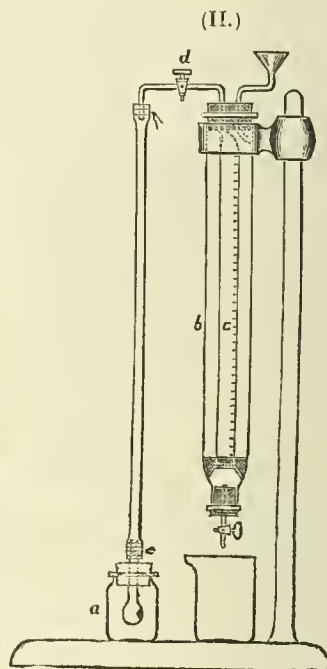
THE form of apparatus shown in the figure is recommended for the fractional distillation of liquids of low boiling-points. A (I.) is a flask holding  $1\frac{1}{2}$  to 2 litres, in which the liquid is placed. It is heated in a bath of water or salt-solution: over the bath is placed the tin-plate cover C, which prevents evaporation of the water, and lessens the danger in case a flask containing an inflammable liquid breaks. B is an empty flask of equal

*New Laboratory Apparatus.* Dingl. J. 254, 67.

*Sampling Instrument.* By H. Angerstein. German Patent 26,680, 1883.—This consists of three parts, as represented in fig. 1 (p. 651): A an iron spindle, pointed below, and terminating above in a forged head; B is a wrought-iron tube, hollowed on its outer circumference for about two-fifths of its length; C is a rod fitting into a hole *c* in the upper part of B. To perform a sampling operation with this instrument, A is introduced into B, and driven into the vessel containing the substance to be sampled by means of a hammer, until B is inserted as far as the protuberance *b*; A is then withdrawn, C inserted into *c*, and by its means B is turned several times in one direction and then withdrawn. The hollowed portion of B contains a stratified sample of the substance.



(I.)



(II.)

size; to it is attached a vertical tube, 65cm. long, and partly filled with broken glass. The two flasks should be 30 to 35cm. apart; and in order to prevent radiations from the hot to the cold one, a sheet of tin-plate D—30cm. broad and 50cm. high—is hung between them.—D. E. J.

*Apparatus for Estimation of Carbon dioxide in Carbonates.* R. Baur. Zeitschr. Anal. Chem. 23, 371.

THIS is an apparatus for the rapid execution of technical analyses of carbonates (soda, etc.). The gas is evolved in the vessel *a* (II.), which is connected by an indiarubber tube, with a three-way cock *d*, and the calibrated measuring-tube *c*; this latter is surrounded by a wider tube *b*, so that the temperature of the gas may be regulated by means of a stream of water. During the evolution of the gas the pressure in *c* is reduced: before measuring off the volume of the gas, the pressure of the gas in *c* is made equal to that of the external atmosphere by altering the level of the liquid in the external tube.—D. E. J.

*Separating Funnel.* By Currier. *American Druggist*, 116, 25.—This simple device (fig. 2) p. 651, consists of an ordinary funnel (*f*) fitted below with a cork (*c*), through which passes a glass tube (*g*) closed above, but containing lateral opening. By raising the tube through the cork this opening becomes a passage for the liquid in the lower portion of the funnel, the flow of which may be stopped at pleasure by lowering the tube until the opening is covered by the substance of the cork.

*Condensing Funnel.*—V. Meyer suggests (Berl. Ber 1883, p. 3000) the suspension of a funnel of the form figured in the annexed drawing (fig. 3), p. 651, over the surface of liquids undergoing evaporation; acids and other volatile constituents are thereby condensed and collected in the recurved rim of the funnel, and may be from time to time removed.

*Temperature Regulator.*—V. Meyer describes (Berl. Ber. 1884, p. 478) a sensitive form of regulator, the essential portion of which is represented in fig. 4. It consists of a brass tube M, 30 centimeters in length, firmly supported in the chamber whose temperature is required to be regulated, into which is inserted the

glass rod G. M carries above, the fulcrum of the lever H, which rests at a distance of five millimètres, upon a horizontal peg carried by G, or, rather, by the cap which surmounts it. Through this cap works the screw R, the lower end of which can be brought into

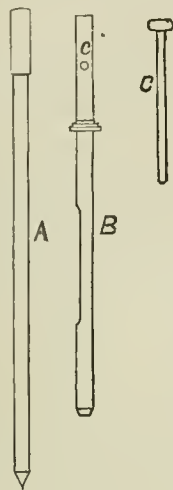


FIG. 1.

contact with the extremity of G. This arrangement enables the lever to be brought to the horizontal position after having suffered displacement as the result of the differential expansion of M and G. The lever H is graduated in Cm., and carries on its longer arm a chain, to which is attached the upper portion of the Kemp-Bunsen gas regulator (figs. 5 and 6). In this apparatus

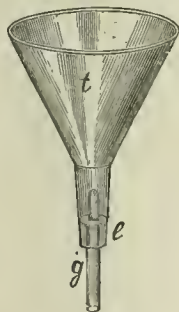


FIG. 2.

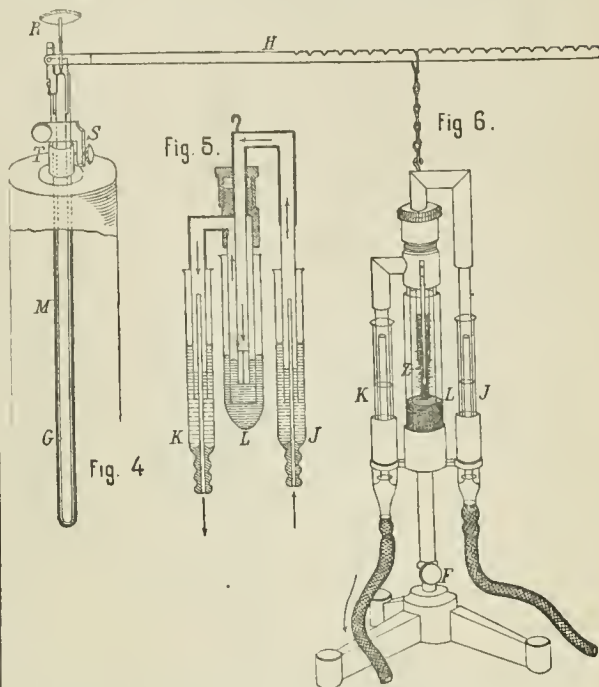


FIG. 3.

the gas takes the course indicated by the arrows. The arrangement and mode of using the regulator is otherwise sufficiently explained by the figures. The following numerical details show the degree of sensitiveness of the instrument. The differential expansion of M G is 0.003mm. per 1°. By the device of the lever this is multiplied in the ratio 5 : 400, i.e., by 80, and the slit which gives exit to the gas in L is raised 0.24mm. out of the mercury. This slit is now 30mm. by 0.2mm., and with the particular air-bath used by the author in his experiments, an exit of gas along its entire length gave a supply sufficient to maintain the temperature of the bath at 300°; each degree of temperature therefore corresponded to a difference in submergence of the slit of 0.1mm.

**Titration Apparatus.**—E. Grenier has devised (German Patent 26, 830, 1883) the burette and accessories represented in figs. 7 and 8. A is the reservoir of standard solution, with which the burette B may be filled by simply turning it to the right. A further turn cuts off the communication between A and B, and on turning yet

further, the flow from the burette commences. The inventor adds a special scale to the burette, which enables the operator to read off in percentage value.



**Burette.**—R. Hübner has patented (German Patent 27,505, 1883) the burette represented in fig. 9. This

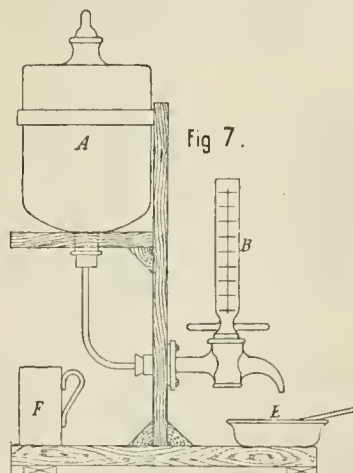


Fig 7.

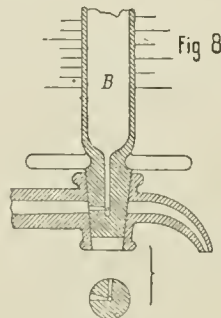
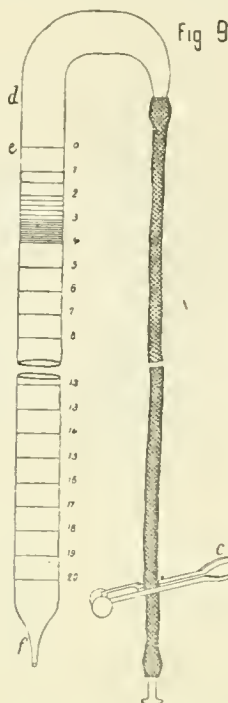


Fig 8

is filled by means of suction, and the efflux is controlled by the pinch cock C.—A. G. Martin (German Patent



26,920) describes the device represented in fig. 9A for the more exact reading off of liquid levels. It consists simply of a coloured line *a* on a white ground *b*.



*The Lathering Point in Hardness Determination.*—This is more sharply determined than by the ordinary.



FIG. 9A.



FIG. 10.

method (shaking bottle) by means of a device introduced by G. Loges (Chem. Ztg. 1884, 69). It consists of a



FIG. 11.

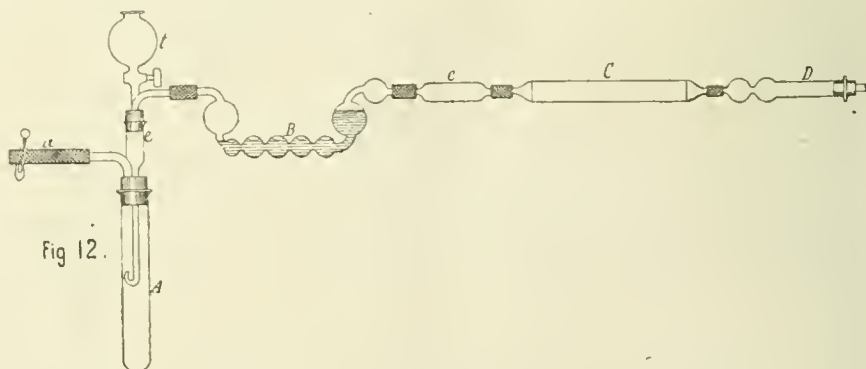


Fig 12.

brass tube (fig. 10) 15cm. long by 4mm. diameter, | by means of a second lamp. The vaporised petroleum  
terminating in a hollow sphere of 1cm. diameter, | passes through the cock *c*, both by way of *c* to feed the

pierced with about 30 small holes. A strong and regular stream of air is blown through this instrument of which the ball is submerged in the solution to which the soap solution is being added. It is found convenient to take 40cc. of the solution under investigation in a vessel of 2-3cm. diameter. Excess of the soap solution is sharply indicated by the sudden rising of the froth in large bubbles to the top of the vessel.

*Modification of Scheibler's Carbonic Acid Apparatus.*—G. Loges employs, for the decomposition of the carbonates, the apparatus represented in fig. 11. The substance is introduced into the outer bottle, the acid into the inner vessel *g*, which is provided with a lateral opening. After the apparatus is put together in the ordinary way, the acid is mixed with the substance by inclining the bottle, and allowing it to flow out by way of *c*.

*Determination of Carbon in Iron and Steel.*—N. B. Wood (Scientific American Suppl. 1883, p. 6553) employs for this purpose the apparatus figured in the annexed drawing (fig. 12). The weighed specimen is introduced

fig 13



into A, where it is slowly decomposed by acid, added from the funnel *t*. The evolved gases, after traversing *l*, which is filled with pumice moistened with sulphuric acid, passes through the bulb tube B containing an alkaline lead solution, and by way of the tube *c* filled with calcium chloride, to the combustion tube C, from which the products of combustion pass to the ordinary absorption apparatus. The "free carbon" is afterwards isolated from the contents of A by filtration.

A. B. Clemence (Engineer, Vol. 56, 387) dissolves 3gr. of the specimen in 130cc. of a solution containing 36gr. cuprammonium chloride, and filters the solution through a platinum funnel, fig. 13, containing an asbestos plug at E. After washing and drying at 150°, a supply of oxygen is admitted at *c*, the other end *a* of the platinum funnel being suitably connected with an absorption apparatus. On applying a gas flame at E the carbon burns, and the carbonic acid is absorbed, and weighed with the usual precautions.

*Decomposition of Minerals by Chlorine.*—R. Schelle (Berg. in Hüt. Ztg. 1883, 589) employs for this purpose the apparatus figured in the annexed ent (fig. 14). It requires no further description beyond noting that the arrangement of stop cocks at *p* and *q* and *r* has the object of allowing the air to be expelled by the chlorine in either part of the apparatus separately.

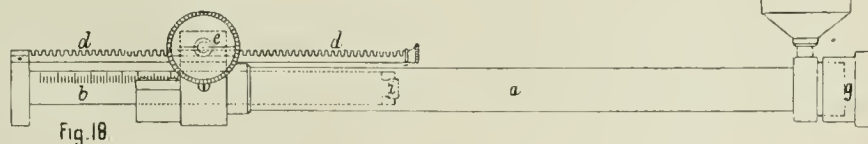
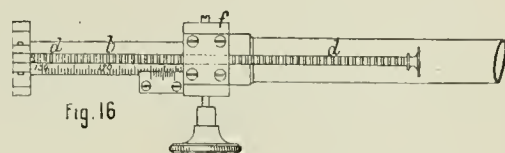
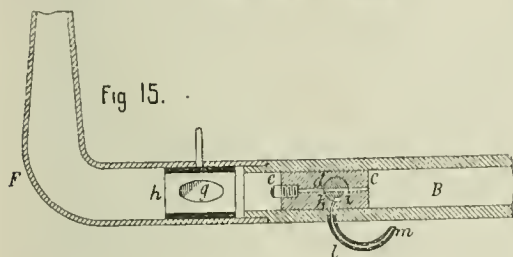
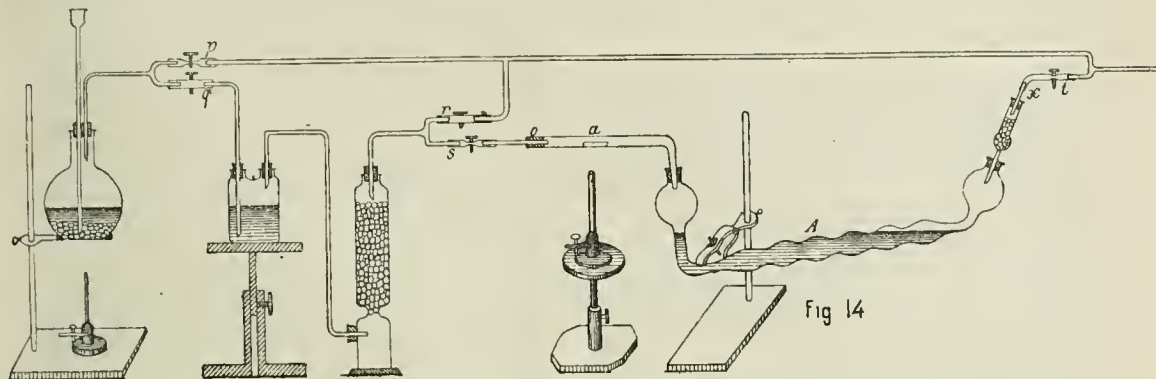
*Lamp for Burning Light Petroleum.*—C. Lilienfein (Stuttgart) constructs the lamp represented in fig. 15 for burning petroleum for laboratory purposes. The actual burner B is screwed into the reservoir of petroleum (not shown), and is fed by the capillary action of the wick which it contains. The lamp is started in the first instance by warming the end of B nearest the reservoir

main flame, and of *i* to reach *m*, where it feeds a small flame which heats B, and so maintains the supply of vaporised petroleum. A full description of the lamp will be found in the *Zeitschr. f. Anal. Chem.* 1884, p. 35.

*Pressure Regulator for Distillation under Diminished Pressure.*—See this Journal, Vol. III. [11], 581.

*Improved Polarimeter.*—F. Schmidt and Hänsch have devised (German Patent 25,439, 1883) a modification whereby the length of the column of liquid under observation in a polarimeter may be varied at pleasure. The instrument is represented in figs. 16, 17, and 18. The

in the Public Trade Schools in Leipsic, and the editor of the "*Chemischen Centralblatt.*" He has divided his work into two sections, the first represented in the 1st volume as the "*Allgemeiner Theil und Niederer Cursus*;" the second, in the 2nd volume as the "*Höherer Cursus.*" The general treatise ("*Allgemeiner Theil*") gives a description of all the apparatus and utensils necessary for instruction in experimental chemistry, and information with regard to their use, making and preparation, cleansing and



glass plate at the right-hand end of the tube *a* is fixed, the plate *i* closes the extremity of the tube *b*, which is movable in and out of *a*, by means of a rack and pinion. In order to perform an observation, the cap *g*, with its glass plate, is removed, and *b* is withdrawn to its fullest extent from *a*, which is then filled with the liquid to be examined. The vessel *h* carrying a short tube below is screwed into *a*, and serves to receive the liquid driven from *a* by the insertion of *b*. By this device observations may be rapidly and easily made upon columns of liquid of varying lengths.

protection. Useful and important rules and precautions are also abundantly given. Information and details as to the erection of laboratories, and construction of draught-places, ventilating-apparatus, etc., are given. The special treatise ("*Besonderer Theil*") describes the methods for carrying out all the lecture-experiments necessary in giving a course of instruction by means of lectures in theoretical chemistry. In the case of each experiment, firstly, the apparatus and reagents are described, and when necessary, details given as to their preparation and testing; and secondly, a description of the experiment itself, and all that is necessary to secure success and avoid mishap. The work commences with an introduction ("*Einleitung*") of 97 pages, giving generally the author's views on the logical method of scientific instruction, and specially his apology (to use an old-fashioned term) for the adoption of his own course of instruction in experimental chemistry. It may well be imagined that a work of this character should have considerable illustration. Accordingly the first volume contains no less than 405 cuts, and the second

## New Books.

TECHNIK DER EXPERIMENTAL CHEMIE. Anleitung zur Ausführung chemischer Experimente beim Unterrichte an niederen und höheren Schulen. Für Lehrer und Studierende; von Dr. Rudolf Arendt. Leipzig: Verlag von Leopold Voss, 1881.

Two volumes in paper covers, large 8vo. size. The author, Professor R. Arendt, is the chemical teacher



374. The whole work being thus illustrated with 779 engravings. The text of the first volume covers 318 pages, and of the second volume, 398 pages. An alphabetical index, covering 11 pages, ends the work. Near the close of Vol. I. tables are given showing with sizes, dimensions, and prices all the paraphernalia necessary for the setting up, arranging, and stocking a chemical laboratory suitable for instruction in chemistry of the classes in elementary schools, according to the German standards. Similar lists and inventories are given at the close of Vol. II., in connection with the requirements of the higher schools.

THE PATENTEE'S MANUAL, being a treatise on the Laws and Practices of Letters Patent, especially intended for the use of Patentees and Inventors, with an Appendix of Statutes, Rules, and Foreign and Colonial Patent Laws, International, Conventional, and Protocol. By JAMES JOHNSON, of the Middle Temple, Barrister-at-Law, and J. HENRY JOHNSON, Solicitor, Assoc. Inst. C.E., past President of the Institute of Patent Agents. Fifth Edition, thoroughly revised, incorporating the Patent Act of 1883. London: Longmans, Green and Company, Paternoster Row; Stevens and Sons, 119, Chancery Lane, 1884.

A LARGE 8vo. volume, bound in cloth, containing 466 pages of subject matter, and a copious index covering 22 additional pages. The table of contents indicates the division of the work into the following sections and chapters:—Table explaining abbreviations. A useful alphabetically arranged indexed table of cases cited throughout the work. Chap. I., a preliminary chapter; II., the subject matter or nature of a patentable invention; III., the incidents of utility and novelty which must by law accompany patentable inventions; IV., who may be a patentee; V., the title; VI., the provisional specification; VII., the complete specification; VIII., opposition to the grant of patents; IX., the patent: its date, duration and extent; X., amendments of specifications; XI., extension or prolongation of letters patent; XII., assignments and licences; XIII., the registration of patents; XIV., infringements; XV., revocations; XVI., offences and their penalties; XVII., international and colonial arrangements; XVIII., the patent office. APPENDIX: Part I., statutes, rules, and forms; Part II., patent laws of foreign countries; patent laws of British colonies; Part III., international convention.

THE ASSAY AND ANALYSIS OF IRON AND STEEL; IRON ORES AND FUEL. By THOMAS BAYLEY, Associate of the Royal College of Science, Ireland, Consulting Chemist, Analyst and Assayer, Birmingham. Author of "A Pocket Book for Chemists." Reprinted from the *Mechanical World*, with additions. Illustrated with 17 wood engravings. London: E. and F. N. Spon, 125, Strand, W.C.; Emmott and Co., 6, York Street, Covent Garden. Manchester: Emmott and Co. New York: E. and F. N. Spon, 35, Murray Street. 1884.

This is a small sized 8vo. volume, bound in cloth, containing a table of atomic weights, recalculated by F. W. Clarke, a table of contents, list of illustrations, and 86 pages of subject matter. The work closes with a table for the analysis of iron ores, and one of factors for calculations. The plan may be gathered from the contents, which are arranged in chapters as follows: Chap. I., preparation of reagents; II., determination of manganese; III., determination of phosphorus; IV., determination of silicon, sulphur and graphite;

V., determination of tungsten; VI., determination of carbon; VII., determination of chromium; VIII., determination of titanium; IX., determination of slag, oxygen, nitrogen, etc.; X., appendix, with selected recent analyses of iron and steel. *Analysis of Iron Ores and Slags*: Chap. XI., the determination of iron; XII., treatment of insoluble samples, and the determination of protoxide of iron; XIII., determination of moisture, combined water and carbonic acid; XIV., determination of silicic, titan and phosphoric acids and sulphur; XV., determination of the bases. *Analysis of Fuel*: determinations of the following: Chap. XVI., moisture and ash; XVII., sulphur; XVIII., coke and volatile matter; XIX., heating power of fuels; XX., analysis of gaseous fuel, blast furnace gases, etc.

## Monthly Patent List.

### ENGLISH APPLICATIONS.

1881.

- 15323 F. J. P. Cheesbrough and E. R. Royston, Liverpool. Improvements in and in means of burning coal and like fuel in furnaces, and in apparatus employed therein for supplying and distributing such coal and like fuel. November 21
- 15337 T. Fox, Junior, Liverpool. Improvements in scouring woollen or worsted cloth or yarn, previous to dyeing. November 21
- 15338 J. Burgess, Liverpool. Improved manufacture of candles. November 21
- 15347 J. Moore, London. Improvements in apparatus for the utilization of waste heat and combustion of smoke in furnaces, grates, and stoves. November 21
- 15365 H. J. Haddan, Westminster. Process and apparatus for electro-plating, and generally applicable for treating metallic surfaces by electrolysis. Communicated by Dr. G. Wagner and Curt Netto, Tokio. November 21
- 15370 A. M. Clark, London. An improved process for the production of substances containing antimony. Communicated by Dittler and Co., Höchst-on-the-Main, through Wirth and Co. November 21
- 15373 H. H. Lake, London. An improved composition chiefly designed for the manufacture of journal bearings. Communicated by J. Schonberg, Friedberg, through Wirth and Co. November 21
- 15393 S. Callard, London. Improvements in the treatment of sulphate of iron. November 22
- 15409 J. Imray, London. An improvement in the manufacture of iron and steel. Communicated by La Société des Acieries de Longwy, Longwy. November 22
- 15412 P. Walker, Edinburgh. The manufacturing of cement Portland tiles. November 21
- 15417 S. Adams, London. Improvements in self-sealing mouthpieces and lids for retorts, stills, and generators, for the production of gas from coal and other substances, also for distillation of liquids and other chemical vapours. Complete specification. November 21
- 15456 W. E. Gedge, London. An improved method of preparing alizarin and other anthracene colouring substances. Communicated by G. Jagenburg, Iydboholm, and Dr. C. Leverkus and Sons, Leverkusen. Complete specification. November 21
- 15466 A. M. Clark, London. An improved process of dyeing. Communicated by A. Bosquet, Paris. November 21
- 15473 H. H. Lake, London. Improvements relating to the production of cold and the manufacture of ice and apparatus therefor. Communicated by W. Raydt, Hanover. Nov. 21
- 15495 J. Sandeman, Glasgow. Improvements in making and applying compounds for preparing or treating textile materials or fabrics of cotton or other vegetable fibres for receiving or retaining colouring matters. November 25
- 15496 A. Mc. Dougall, Penrith, Cumberland. Improvements in the manufacture of ammoniacal salt and in the purification of gas. Complete specification. November 25
- 15497 P. Kirk, Manchester. Improvements in ovens or furnaces for heating ingots or blooms or for maintaining the heat thereof. November 25
- 15501 J. J. Shedlock, Barnet, Hertfordshire. Improvements in the process of and apparatus for the extraction of metals from their ores. November 25
- 15520 H. J. Haddan, London. A continuous kiln for burning limestone, cement, and similar materials. Communicated by A. Dufour, Dôle. November 25
- 15531 W. R. Lake, London. Improvements relating to the purification of fatty or oleaginous substances, and apparatus therefor. Communicated by A. F. Le Myé, Paris. Nov. 25
- 15546 G. Quick, Chipping Campden, Gloucestershire. Improvements in propelling composition and cartridges for ordnance. November 26
- 15549 S. B. Woodhead and W. Baker, Liverpool. Improvements in apparatus for reducing or raising the temperature of liquids. November 26



- 15581 F. W. Lacey, London. An improvement in the purification of sewage by precipitation. November 26
- 15586 E. V. Gardner, London. Improvements in the construction of chambers to be employed in the manufacture of white lead, and in the arrangement of the lead within the same. November 26
- 15587 A. C. Henderson, London. Improvements in the method of and in the process of transforming amylaceous and cellulose matters into saccharose or crystallizable sugar. Communicated by L. Aubert and V. Giraud, Lyon. Nov. 26
- 15599 A. D. Cohen, London. Improvements in disinfectants. November 27
- 15628 G. J. C. Marie, Baron de Liebhaf, London. Colouring stone, marble, and other materials for building and other purposes. May 23 (previously included in No. 8121)
- 15631 W. Cunningham, London. Improvements in lime and brick kilns, and in cement kilns and mills. November 27
- 15614 A. G. Brookes, London. Improvements in metallic alloys or compounds, and in producing the same. Communicated by H. J. F. Niewerth, Hanover. November 27
- 15617 C. W. Curtis, London. Improvements in the Manufacture of explosives. November 27
- 15530 W. R. Lake, London. Improved processes and apparatus for obtaining fibrous material from ramic and other plants. Communicated by J. O'Neil, United States. November 27
- 15634 H. J. Hadden, London. Process and apparatus for treating textile materials with liquids or gases. Communicated by J. Brandt and G. W. von Nawrocki, agents for C. Weber and Co., Thann. November 27
- 15639 J. W. Kynaston, Liverpool. Improvements in or connected with the manufacture of sulphite of calcium. Nov. 28
- 15673 E. Crummack, Manchester. An apparatus for the destruction of sewage and other deleterious gases and germs of disease in sewers. November 28
- 15682 W. Gratrix and J. Best, London. An improved size for manufacturing purposes. November 28
- 15690 A. F. Spaw, San Francisco, United States. Improvements in hand grenades or chemical fire-extinguishing devices. November 28
- 15701 W. Noble, London. Improved means of consuming smoke in steam boiler furnaces. November 28
- 15712 R. Elliott, London. Improvements in explosive projectiles and in the fuses therefor. November 28
- 15719 H. Knight, Liverpool. Improvements in the manufacture of sulphide of zinc or of pigments having sulphide of zinc as a constituent or ingredient. November 29
- 15739 W. G. Margetto, London. Improvements in the manufacture of Portland cement, and in apparatus to be used in the said manufacture. November 29
- 15762 L. J. Pirie and H. Roberts, London. Improvements in the manufacture of dip candles, and in apparatus to be employed in this manufacture. November 29
- 15778 E. Fontenille, Paris. An improved process for refrigerating and congealing liquids. December 1
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